Effects of cooling media on the formation of Martensite-Austenite microconstituent in a HSLA steel

Zayra Moreno-Fabian*, Gregorio Solís-Bravo

Universidad Veracruzana, Faculty of Chemical Sciences, Veracruz, México (*Corresponding author: GS16001394@egresados.uv.mx)

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ABSTRACT: The effect of different cooling conditions on the formation of Martensite-Austenite (MA) in a High - Strength Low - Alloy (HSLA) steel was assessed. The MA constituent is detrimental to impact toughness in pipeline applications, so the purpose of this research was to minimize its presence through the choice of effective cooling media and optimal parameters such as the cooling rate and final cooling temperature. The volume fraction, size and morphology of MA was evaluated by selective etching and corroborated trough SEM and EBSD. Vickers hardness testing agreed with the measured MA volume fractions. The sample cooled with helium gas and salt bath with the lowest final cooling temperature of 460 °C, exhibited a fine mixture of ferritic bainite, granular bainite and the lowest volume fraction of MA, along with MA smaller particle average size. A high cooling rate and a decrease in the final cooling temperature resulted in a decrease in the volume fraction and average particle size of MA.

KEYWORDS: Accelerated cooling; Linepipe steel, Martensite-Austenite (MA); Retained Austenite; Selective etching; Scanning electron microscopy (SEM)

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RESUMEN: Efectos del tipo de enfriamiento en la formación del microconstituyente Martensita – Austenita en un acero de Alta Resistencia y Baja Aleación (HSLA). Se evaluó el efecto de diferentes condiciones de enfriamiento en la formación de la Martensita-Austenita (MA) en un acero de Alta Resistencia y Baja Aleación (HSLA). El componente MA es perjudicial para la tenacidad al impacto en aplicaciones de tuberías, por lo que el objetivo de esta investigación fue minimizar su presencia mediante la elección de medios de enfriamiento eficaces y parámetros óptimos como la velocidad de enfriamiento y la temperatura final de enfriamiento. La fracción de volumen, el tamaño y la morfología de la MA se evaluaron mediante ataque selectivo y se corroboraron mediante SEM y EBSD. Las pruebas de dureza Vickers coincidieron con las fracciones de volumen de MA medidas. La muestra enfriada con gas helio y baño de sales con la temperatura de enfriamiento final más baja de 460 °C, exhibió una mezcla fina de ferrita bainítica, bainita granular y la menor fracción de volumen de MA, junto con un tamaño promedio de partícula menor. Una alta velocidad de enfriamiento y una disminución de la temperatura de enfriamiento final de as partículas de MA.

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PALABRAS CLAVE: Acero para tuberías; Ataque selectivo; Austenita retenida; Enfriamiento acelerado; Martensita-Austenita (MA); Microscopía Electrónica de Barrido (SEM)

ORCID ID: Zayra Moreno-Fabian (https://orcid.org/0000-0002-0478-7117); Gregorio Solís-Bravo (https://orcid.org/0000-0001-6522-6965)

1. INTRODUCTION

High-Strength Low-Alloy steels (HSLA) are an important category of steels; some of their most important applications are the pipeline systems, the most effective method of transporting crude oil and natural gas. Their current designs must consider all aspects of a steel's performance: strength, ductility, and toughness, i.e., a high performance and durability to operate in low temperature, high pressure, and impact environments (Okatsu *et al.*, 2005; Belato *et al.*, 2013; Kabanov *et al.*, 2019). These properties depend on the proper combination of chemical composition, thermo-mechanical processing, and accelerated cooling conditions (Zhao *et al.*, 2002).

An undesirable defect in pipeline steels is the presence of the microconstituent Martensite-Austenite (MA), which has been linked with the reduction of impact toughness because they are stress concentrators and facilitate crack propagation (Reichert *et al.*, 2012; Huda *et al.*, 2016; Wang *et al.*, 2017; Takayama *et al.*, 2018). Therefore, the presence of MA constituents in the final microstructure leads to a decrease in steel ductility (Reichert et al., 2012). It is known that the mechanism of MA formation is the austenite decomposition which is controlled by the cooling stage. The formation of acicular ferrite, aided by accumulated deformation in austenite has been shown to reduce the formation of MA (Zhao et al., 2002; Zhao, 2016; Kabanov et al., 2019). However, during cooling, the parameters that influence their formation are not as clear. Research has found that cooling rate is the main factor, while others suggest that the variable that influences the most is the final cooling temperature (Cota and Santos, 2000; Wang et al., 2017).

Therefore, the goal of the present study was to minimize the MA formation and obtain a bainitic microstructure in a microalloyed HSLA steel through designing the cooling stage: molten salt bath for isothermal cooling, helium gas for continuous cooling and a combined cooling route. Quantitative relationships between cooling variables and fraction, morphology and size of MA particles were investigated using optical microscopy (OM), scanning electron microscopy (SEM), electron back-scattered diffraction (EBSD) and Vickers hardness testing.

2. MATERIALS AND METHODS

The chemical composition of the steel used in this investigation is given in Table 1. This steel contains Nb, Ti and V as microalloying elements. Mo improves fatigue resistance and Mn enhances weldability. This steel was developed as a commercial grade and received in the as hot rolled condition.

Specimens (15x9x15 mm) were cut. For precise temperature measurement and data collection of the cooling curves, K-type thermocouples were welded to the surface of each sample and connected to a digital thermometer.

The samples were heated to 1100 °C, within the austenitic region and above the no-recrystallization temperature, Tnr = 1008 °C, in a Carbolite muffle furnace for 5 min. The no-recrystallization temperature was calculated using the Boratto equation (Konca, 2020):

$$Tnr = 887 + 464*C + (6645*Nb - 644\sqrt{Nb}) + (723*V - 230\sqrt{V}) + 890*Ti + 363*Al - 357*Si$$
(1)

Then, samples were cooled inside the furnace to 880 °C to simulate an approximate final rolling temperature and this was followed by different interrupted accelerated cooling paths designed through the CCT diagram (Fig. 1) of this steel, which was generated through the free version of JMatPro software by entering the chemical composition, austenitizing temperature and austenitic grain size of the microalloyed.

To obtain a bainitic microstructure with better properties, finishing cooling temperatures below the bainitic nose (524 °C) were selected for the complete transformation of bainite.

The chosen cooling media were molten nitrate salts for isothermal transformation and helium gas for continuous transformation. A route with combined cooling media was proposed, helium at a higher cooling rate to avoid ferrite transformation and salts for complete bainite transformation. The

TABLE 1. Chemical composition of steel (wt. %). Fe to balance.

С	Mn	Si	V + Ti + Nb	P + S	Cu + Ni + Cr	Al	Ν	Ca
0.05	1.5	< 0.30	< 0.25	< 0.02	< 0.65	< 0.05	< 0.008	< 0.0015

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FIGURE 1. Schematic representation of the simulated heat treatment for the steel investigated.

weight of the salt mixture for each test was 200 g and was melted in a Felisa muffle furnace, model AR 340. The salts were sustained at 500 °C and 450 °C. The cooling rate and the final cooling temperature with helium gas were manually controlled by means of a pressure valve (1.79 MPa max.), which allowed a regulation of the helium flow rate. Each experimental process is detailed in Table 2.

After the interruption of the accelerated cooling, the samples were cooled down to the room temperature. The schematic representation of the thermal cycles is shown in Fig. 2.



FIGURE 2. Continuous cooling transformation (CCT) diagram of microalloyed HSLA steel obtained with JMatPro software.

2.1. Microstructural characterization

Samples were carefully prepared following the standard procedure. The roughing of the samples was conducted with abrasive grits 400, 600, 800, 1000 and 1200 using water cooling. Subsequently, the sample was polished with alumina suspension and diamond paste.

For the identification of the final microstructure, the samples were etched with 3% Nital (1.15 ml HNO₃ in 25 ml C₂H₅OH) with a rubbing time of 3 to 5 seconds and analyzed at 400x by OM. However, the presence of MA could not be identified in all samples with this etching and magnification due to their small size. Therefore, they were etched with sodium metabisulfite solution (10 g $Na_2S_2O_2$ in 100 ml distilled water) with immersion time of 10 seconds. and analyzed at 800x by OM. Sodium metabisulfite reveals white MA constituents in a brown matrix. Six images were captured for each sample with an optical microscope VanGuard. ImageJ software was used for transforming the images to binary and quantify the volume fraction and MA manual delimitation of the MA islands was utilized for measuring average Feret diameter of the particles and estimate the average size. The MA morphology was investigated using a Scanning Electron Microscope (SEM) at the magnifications of 2500x and 5000x. The samples were etched with 3% Nital for 3 seconds

With the intention of corroborating the volume fraction of MA using a second technique, Electron Backscatter Diffraction (EBSD) was used to acquire Image Quality maps. For this technique, samples were hand-polished with alumina suspension (1 µm, $0.3 \ \mu m$ and $0.05 \ \mu m$), then were vibropolished for 3 h with alumina slurry (0.05 μ m) at a frequency of 80 Hz and finally, they were cleaned and immersed with ethanol in a beaker for ultrasonic cleaning for 5-10 min. This technique was performed in a Jeol Scanning Electron Microscope with a Field-Emission Gun (FEGSEM) using a 20 kV and 13 nA beam in a hexagonal pattern with 0.2 µm step size and BCC iron as a reference. These conditions make particles like MA and carbides show relatively low IQ values, offering a contrast with the matrix not possible with SE imaging. Distinction of MA particles utilized this contrast, particle morphology and

TABLE 2. Selected parameters for each experimental process

Sample	Reheating °C	T _{sc} °C	T _{FC} ^o C	Transformation	Cooling media
А	1100	880	520	Isothermal cooling	Nitrate salts
В	1100	880	470	Isothermal cooling	Nitrate salts
С	1100	880	520	Continuous cooling at 9.5 °C·s ⁻¹	Helium gas
D	1100	880	460	Continuous cooling at 20 °C·s ⁻¹ and isothermal cooling	Helium gas and nitrate salts

location. These measurements were done by manually circling MA particles in the IQ maps using ImageJ software.

Finally, to validate the microstructural results, Vickers microhardness was measured using a Mitutoyo, model HM-125 microhardness tester, with load of 30 g.

3. RESULTS

3.1. Cooling curves

Analysis of the cooling curves provides useful information about the undergoing transformations, when compared to the CCT diagram. The cooling curves of each experimental process are shown in Fig. 3 in overlay with the CCT diagram presented in Fig. 2. All the cooling conditions begin transformations with ferrite formation, followed by bainite formation. Only sample C appears to not fully undergo bainitic transformation. Nevertheless, the bainitic transformation is a different process for every condition. It is observed that the nitrate salts (sample A and B) did not allow the start of bainitic transformation to be reached in a short time that would allow its complete transformation in an isothermal manner, therefore the cooling rate was calculated above and below the Bs for a better interpretation of the results. According to the results reported in Table 3, as the cooling rate (\dot{T}) increases and the finish cooling temperature (T_{FC}) decreas-



FIGURE 3. Schematic representation of the cooling routes of microalloyed HSLA steel.

TABLE 3. Analysis of cooling routes for microalloyed HSLA steel

Sample	T _{FC} °C	Bs °C	<i>Ť</i> (880°C-Bs) °C·s⁻¹	<i>Т</i> ́ (Bs-T _{FC}) °С∙ s ⁻¹
А	520	570	16.3	0.33
В	470	550	36.6	1
С	520	590	9	11.6
D	460	550	36.6	5.6

Cooling rates are approximated

es, the start bainite temperature (Bs) is lower and the time required for its complete transformation is shorter.



FIGURE 4. Optical micrographs (400x) for the samples cooled in different media and finish temperatures: (a) Nitrate salts-520 °C, (b) Nitrate salts-470 °C; (c) Helium gas-520 °C; (d) Helium gas and nitrate salts-460 °C. (BF: bainitic ferrite; PF: polygonal ferrite; GB: granular bainite; MA: MA constituent).

3.2. Final microstructure

Most microstructural components could be identified through OM and 3% Nital etching. For better contrast of MA component, sodium metabisulfite aqueous solution was used as etchant. It can be seen from Fig. 4a that the microstructure of sample A associated with a high finish cooling temperature and an intermediate cooling rate, consists mainly of bainitic ferrite (BF), granular bainite (GB) and polygonal ferrite (PF) grains. By contrast, the microstructure of sample B (Fig. 4b), consists of bainitic





FIGURE 5. Optical micrographs (800x) for the samples cooled in different media and finish temperatures: (a) Nitrate salts-520 °C, (b) Nitrate salts-470 °C; (c) Helium gas-520 °C; (d) Helium gas and nitrate salts -460 °C. Pre-etched in 3% Nital and etched with a solution of sodium metabisulfite 10%. MA particles analyzed with ImageJ.

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ferrite with wider laths and granular bainite with a slight presence of fine-grained polygonal ferrite.

For the sample C with a high finish cooling temperature and a slow cooling rate (Fig. 4c) a mixture of polygonal ferrite and grains of bainitic ferrite with granular bainite is observed. Here, grain size and BF lath size are relatively large, because of temperature and prolonged processing time. Some MA constituents are visible in white color. Finally, for the sample D (Fig. 4d) associated with the lowest final cooling temperature and highest cooling rate, higher fraction of bainitic ferrite with fine laths and a lower fraction of granular bainite are observed. In all samples, carbides were observed at the boundaries of granular bainite and between upper bainite laths.

3.2.1. Martensite – Austenite (MA)

For the analysis of MA constituents, it is important to know their volume fraction, size, and morphology, which were analyzed at 800x using OM (Fig. 5) on specimens etched with sodium metabisulfite aqueous solution, the results are reported in Fig. 6.

The volume fraction of MA in sample A (Fig. 5a) is 0.0438 with blocky MA islands ranging in size

from 6 to 14 µm and elongated MA islands of 4 to 8 μ m. The average MA particle size is 4.34 μ m with a standard deviation of 0.2432. While for sample B (Fig. 5b) with a finer BF microstructure and lower GB fraction, the MA volume fraction (0.0266) and average particle size of 3.9 μ m (σ = 0.4887) decreases. In the case of sample C, continuously cooled to the same temperature as sample A, it reports elongated MA particles with sizes ranging from 6.4 µm to 9.9 µm located between the BF laths and fine particles randomly distributed in the matrix. The average volume fraction is 0.0352 with an average MA particle size of 3.75 μ m ($\sigma = 0.3349$) being the second sample with the largest MA particle size. Finally, sample D (Fig. 5d), has the lowest volume fraction and average MA size of 0.0234 and 3.7 μ m (σ = 0.1959). More detailed observations of MA islands morphology were obtained using SEM (Fig. 7)

In sample A (Fig. 7a), where the high-temperature transformation products are mostly PF, GB and coarse BF, the MA constituents appear as large islands or blocks between the grain boundaries (of PF) and within the grain (of GB). In sample B (Fig. 7b), there are MA constituents of elongated and thin morphology between BF laths as well as fine particles in GB grains.



FIGURE 6. Influence of cooling parameters on the formation of MA: (a) Average volume fraction; (b) Average size.

TABLE 4. Results of cooling routes for microalloyed HSLA steel

Sample	T _{FC} ⁰C	Bs ⁰C	<i>İ</i> (880°C-Bs) °C∙s¹	<i>τ</i> (Bs-T _{FC}) °C·s ⁻¹	Microstructure	$f_{\scriptscriptstyle m MA}$	HV
А	520	570	16.3	0.33	BF + PF + GB	0.0438	219.31
В	470	550	36.6	1	BF + GB	0.0266	215.19
С	520	590	9	11.6	BF + PF + GB	0.0352	213.74
D	460	550	36.6	5.6	BF + GB	0.023	204.92

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d)

FIGURE 7. SEM images for the samples cooled in different media and finish temperatures: (a) Nitrate salts-520 °C, (b) Nitrate salts-470 °C; (c) Helium gas-520 °C; (d) Helium gas and nitrate salts-460 °C

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For sample C (Fig. 7c), elongated and large MA particles are observed in comparison to sample B, between the BF laths, as well as irregularly morphology particles at the grain boundaries. Finally, in sample D (Fig. 7d), a combination of elongated and round islands particles is observed at the grain boundaries.

As an additional analysis, the aspect ratio and roundness of the MA particles for each sample were investigated by ImageJ (Fig. 8). However, these results did not show considerable contrast. Hence, the different cooling paths did not influence morphology as much as they influenced volume fraction.

Utilizing Image Quality (IQ) maps through EBSD, the MA constituent presence was confirmed. Due to limited availability of the equipment, only the samples with higher and lower average volume fraction were analyzed with this technique. On the maps shown in Fig. 9, the presence of small islands of highly distorted crystal associated with MA can be observed. The MA volume fraction was measured, results were compared with the fractions obtained using OM (Fig. 10). The presence of these MA islands in the sample A (cooled in nitrate salts with T_{FC} =520 °C) is higher than that observed in the sample D (cooled with helium and nitrate salts with T_{FC} =460 °C) with a volume fraction of 0.0585 and 0.0435, respectively, agreeing with the appearance and quantification of the micrographs taken by OM.

The utilization of OM, SEM and EBSD had a synergistic effect on the reliable identification and assessment of MA. Each technique offered certain advantages. OM relies on selective etching and offers good contrast, this technique was good for surveying vast areas and having a representative sample, however, resolution is limited, so smaller particles (<0.5 um) may be overlooked. SEM can improve resolution, but contrast is reduced, the MA particles' location and morphology need to be known beforehand. EBSD-IQ maps offer better resolution and contrast, giving the best opportunity to identify MA presence, location, and morphology. However, there is uncertainty as to whether dark particles are carbides, MA or just retained austenite. Hence, authors combined these techniques and learn to identify MA particles (location, shape, color) before quantifying.







FIGURE 9. Shape description of the MA particles of the samples cooled in different media and finish cooling temperatures: (a) Aspect Ratio (b) Roundness



Figure 10. Volume fraction of MA by different analysis techniques of the samples cooled in different media and finish temperatures (a) Nitrate salts-520 °C; (b) Helium gas and nitrate salts-460 °C.



Figure 11. Vickers microhardness of the samples cooled in different media and finish temperatures: (A: Nitrate salts-520 °C; B: Nitrate salts-470 °C; C: Helium gas-520 °C; D: Helium gas and nitrate salts-460 °C).

Finally, the hardness variations among the phases present in the final microstructure of the samples were evaluated and quantified. According to Fig. 11, the sample A with the highest MA volume fraction exhibited the highest microhardness values, while the sample D with the lowest MA volume fraction reported the lowest microhardness values.

4. DISCUSSION

The final microstructure of the samples shows that with an increase in the cooling rate, the amount of polygonal ferrite and granular bainite decreases while the volume fraction of bainitic ferrite with fine laths increases. Due to the increase in the cooling rate, the reduction in the Bs temperature leads to an increase in the driving force for the nucleation rate of sub-units of ferrite. Consequently, a higher amount of transformed bainite and a lower volume fraction of retained austenite are obtained. These results are studied and confirmed in several investigations (Zhao, 2016; Rodrigues *et al.*, 2000; Konca, 2020).

The highest volume fraction of MA corresponds to the sample A, which is a consequence of a favorable carbon diffusion to the residual austenite during the transformation time at high temperatures (TFC=520 °C). Contrastingly, the sample C with the same finish cooling temperature has also a high average fraction of MA, but the average MA size is smaller because in continuous cooling, the diffusion of carbon is less favored than during isothermal stage Furthermore, in the following air subcooling, the transformation is completed at lower temperatures, further decreasing the possibility for C diffusion. The sample that obtained the lowest average volume fraction of MA was the sample D with combined cooling and $T_{FC} = 460$ °C. The minimization of the MA volume fraction is attributed to a fast rate for the start of the bainitic transformation, a brief time for the complete transformation and a low final cooling temperature. These are considered optimal conditions to limit the carbon diffusion and, thus, minimize MA in the final microstructure. Other research agrees with these results, (Biss and Cryderman, 1971; Cota and Santos, 2020). It has been shown that if the cooling rate, and thus the transformation rate, is slow enough, the carbon can diffuse away from the advancing ferrite-austenite interface and the austenite is then enriched in carbon. However, if the cooling rate is increased, there is less time for the diffusion of carbon in austenite, hence, much higher carbon contents and steeper carbon gradients develop at the austenite-ferrite interface. The work of Huda et al. (2016) indicates the opposite; however, it is a different thermal route.

The MA constituents were located between the laths of the BF and near the grain boundaries with two dif-

ferent morphologies: elongated rod and blocky shape. When the main transformation product was upper bainite, i.e., a microstructure with parallel lath packages, the retained austenite existed mainly between the laths, giving place to the elongated rod shapes of the MA constituents, while the blocky morphology, was found next to the grain boundaries. As discussed above, the size of these particles depends on the cooling path, which determines the carbon enrichment. Due to this carbon enrichment of the residual austenite, the microhardness of the MA is remarkably high relative to the matrix (Rodrigues et al., 2000). Therefore, the sample A with the highest MA volume fraction reported the highest hardness value (219.3 HV), while the sample D with the lowest MA volume fraction reported the lowest value (204.9 HV), confirming the validity of the microstructural results.

Based on the above, it is established that the formation of MA constituents is a carbon diffusion-controlled process, leading to carbon enrichment in austenite. According to (Zhao *et al.*, 2002; Liang *et al.*, 2014), MA formation can be described in two stages: carbon diffusion in austenite above the start of bainitic transformation (Bs) and carbon partitioning between bainitic-ferrite and austenite at low temperatures. The present results agreed with these known mechanisms.

5. CONCLUSIONS

The effect of different cooling routes, on the formation of MA constituents in the final microstructure of a microalloyed HSLA steel, has been investigated. Microstructure characterization was obtained through selective etching, Optical Microscopy and SEM, then corroborated by EBSD Image Quality maps and Vickers microhardness testing. It was found that, at low cooling rates of 9 °C·s⁻¹ and 16.3 °C·s-1 with high finish cooling temperatures of 520 °C, the microstructure consists of polygonal ferrite, bainitic ferrite and granular bainite. In contrast, as the cooling rate is increased (36.6 °C·s-1) and finish cooling temperature is decreased (470 °C and 460 °C), the bainite laths and phase fraction of PF and GB decreased. At low cooling temperatures, carbon diffusion is limited, therefore, the MA volume fraction decreases. Whereas, at high finish temperatures, diffusion is favored towards residual austenite, forming a higher MA fraction with large particle size. MA constituents can be distinguished between bainitic laths within grains and at grain boundaries, with island or blocky and elongated morphologies. Morphology does not appear influenced by the different cooling processes.

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