A Transmission Electron Microscopy Investigation of Reaustenitized-and-Cooled HSLA-100 Steel

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Abstract A continuous-cooling-transformation diagram for 0.06% C HSLA-100 steel (austenite grain size of about 10 μm) was reexamined in combination with hardness data and light-microscope images. Data suggested the presence of a martensitic microstructure over a wide range of cooling rates. A specimen cooled at approximately 17 $^{\circ}\text{C/s}$ was chosen for detailed microstructural investigation. Transmission electron microscopy revealed a microstructure consisting primarily of martensite laths with a moderate-to-high dislocation density. About 5% of retained austenite was present as interlath films. An orientation relationship close to Kurdjumov–Sachs was verified for martensite and austenite, and adjacent laths exhibited an axis/angle pair consistent with two variants of this relationship. Some regions of twinned martensite were observed, and multi-variant cementite precipitation was observed within large autotempered martensite laths. All results are consistent with this steel in its as-received mill-processed condition.

Keywords HSLA-100 · TEM · Martensite · Retained austenite · Electron diffraction

Introduction

Wilson et al. [1] examined the microstructures and mechanical properties of several low-carbon copper-precipitation-strengthened alloys, including HSLA-80, HSLA-100, and two

alloys of “intermediate” compositions.¹ This “class” of steels was developed to replace steels with similar strengths, but notably higher carbon content [2, 3]. A key reason for the development of this class of steels was to achieve improved weldability by lowering the carbon content but maintaining high strength through copper-precipitation hardening. The steels studied by Wilson et al. [1] were processed in a commercial mill by rolling to plate followed by a reaustenitize-and-quench step. Each steel was microalloyed with niobium, and all steels possessed an equiaxed fine-grained austenitic microstructure (e.g., about 10 μm) prior to the final mill quench.

The microstructure of HSLA-80 steel was *shown* to contain primarily fine-grained polygonal ferrite over a wide range of cooling rates. Transformation of austenite to polygonal ferrite occurred at relatively high temperatures (i.e., between about 550° and 800 $^{\circ}\text{C}$, depending on cooling rate), with final ferrite grain sizes of 5 μm or larger. Subsequent work [4] revealed a low dislocation density within the grains of polygonal ferrite, and secondary microconstituents that included carbon-enriched islands (a few micrometers in size) of bainite with coarse carbides, martensite, and some austenite.

It was *concluded* that an HSLA-100 steel with 0.06% C exhibited a martensitic microstructure over a wide range of cooling rates. Two alloys of “intermediate” compositions displayed a microstructure referred to as “granular bainite” over a wide range of cooling rates. The latter microstructure consisted primarily of small crystals of ferrite (a few micrometers in size) with both equiaxed and non-equiaxed morphologies and moderate dislocation density [5]. Additionally, the microstructure also contained a small amount

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¹ HSLA steel refers to high-strength low-alloy steel. The numerical designations indicate the minimum acceptable yield strength in units of ksi.

of carbon-enriched, sub-micron-sized “blocky” islands of martensite and retained austenite (M/A constituent).

The term “granular bainite” was used based on work by Habracken and Economopoulos [6] published in the symposium proceedings *Transformation and Hardenability in Steels*. Since this article was published, other terms have been suggested for “granular bainite” such as carbide-free bainite, acicular ferrite, granular ferrite, and others. In the remainder of this article, this microstructure will be referred to as acicular ferrite [5].

Wilson et al. [1] demonstrated that the yield-strength/Charpy-impact-toughness behavior of the HSLA-100 steel with a martensitic microstructure was superior to that of the HSLA-80 steel with a polygonal ferrite microstructure. The strength/toughness behaviors of the two steels with “intermediate” compositions (and acicular ferrite microstructures) were intermediate to those of the HSLA-80 and 100 steels. In addition, a second HSLA-100 steel with a comparatively low-carbon content of 0.04% C exhibited an acicular ferrite microstructure in 29-mm-thick plates, with concurrently poorer strength/toughness behavior, as compared with the 0.06% C HSLA-100 steel [1].

While the microstructures of the HSLA-80 steel and one of the “intermediate” compositions studied by Wilson et al. [1] have been documented in detail [4, 5] via transmission electron microscopy, the microstructure of the 0.06% C HSLA-100 steel has not, until recently. The original conclusion that the latter alloy possessed a martensitic microstructure over a wide range of cooling rates was strongly supported by several important observations. Light microscopy showed that the microstructure of 0.06% C HSLA-100 steel was notably different than that of the “intermediate” alloys (as well as the 0.04% C HSLA-100 steel), thereby suggesting that the structure was something other than acicular ferrite with M/A. The hardness of the 0.06% C HSLA-100 steel was close to that expected for as-quenched martensite based on carbon content [7], unlike 0.04% C HSLA-100 steel and the “intermediate” alloys. Also, a continuous-cooling-transformation (CCT) diagram (from the 0.06% C HSLA-100 steel, see Fig. 1) showed a well-defined martensite-start temperature (M_s) of about 425 °C over a wide range of cooling rates [1] that agrees with the calculated value from an empirical equation of Andrews [8]. A recent study via transmission electron microscopy showed that the 0.06% C HSLA-100 steel processed exclusively in a plate mill possessed a “fully” martensitic microstructure [9].

In two studies published after the work by Wilson et al. [1], the major thrust was to examine fine microstructural features via transmission electron microscopy of specimens that came directly from CCT studies [4, 5]. Cylindrical test specimens were reaustenitized to 905 °C and cooled at various rates in a Gleeble® 1500 thermo-mechanical simulator [10]. Hence, the purpose of this study is, in essence,

Fig. 1 Continuous-cooling-transformation diagram for 0.06% C HSLA-100 steel. Prior austenite grain size approximately 10 μm . Labels are A (austenite), M (martensite), AF (acicular ferrite), GF (granular ferrite), and PF (polygonal ferrite)

to complete a trilogy of studies that includes HSLA-80 steel, an “intermediate” A710-type steel, and 0.06% C HSLA-100 steel. In each of the earlier papers [4, 5], transmission electron microscopy results emphasized the most-prevalent microstructure, i.e., the microstructure that formed over a wide range of industrially important cooling rates. Collectively, these papers show a progression of the predominant microconstituent from polygonal ferrite (HSLA-80), to acicular ferrite (“intermediate” A710-type compositions), to martensite (HSLA-100) as alloy level increases but with a common austenite grain structure (equiaxed grain size about 10 μm).

Low-carbon copper-containing steels discussed above are put into service after a high-temperature (usually above 600 °C) aging heat treatment [1]. This treatment produces steel with an excellent combination of strength and toughness. While a high temperature is needed to induce copper precipitation, other processes also occur, most notably the recovery of dislocation substructure and elimination of low-angle boundaries. As discussed above, martensite has superior mechanical properties compared to acicular ferrite, and these microconstituents also have important differences in age-hardening behavior. Thus, the purpose of this article is to document the fine-scale microstructural features of a 0.06% C HSLA-100 steel in the as-quenched condition that was produced from undeformed, fine-grained austenite. A key aspect of this study will be to assess whether or not the observations agree with generally accepted features of lath martensite.

Experimental Procedures

The reported chemical composition of the steel examined was (in wt.%): 0.06 C, 0.83 Mn, 0.010 P, 0.002 S, 0.37 Si, 1.66 Cu, 3.48 Ni, 0.58 Cr, 0.59 Mo, 0.028 Nb, 0.028 Al, balance Fe. The steel was received in the form of a

29-mm-thick (i.e., 1 and 1/8 inch) plate that had been reaustenitized and quenched. This is the same steel that was studied in Ref. [1, 9, 10].

Test specimens were machined to a length of about 102 mm and a diameter slightly larger than 6 mm, and possessed a 12-mm-long reduced section of 3 mm in diameter, with a 30- μm surface polished finish. These specimens were heated from room temperature at a rate of 3 $^{\circ}\text{C/s}$ to 905 $^{\circ}\text{C}$, held for 300 s, and cooled at various rates in a Gleeble[®] 1500 thermo-mechanical simulator. The cooling programs were of a Newtonian nature [10, 11], and a simple estimate of cooling rate was determined by dividing 300 $^{\circ}\text{C}$ (i.e., 800–500 $^{\circ}\text{C}$) by the total time that the specimen cooled in this 300 $^{\circ}\text{C}$ -temperature interval (referred to as $\Delta t_{800 \rightarrow 500}$).

To determine the temperature range over which austenite forms during reheating, a heating rate of about 0.03 $^{\circ}\text{C/s}$ (i.e., 2 $^{\circ}\text{C/min}$) was employed. The temperatures at which austenite begins to form (A_{c1}) and at which a fully austenitic microstructure is obtained (A_{c3}) during heating were determined from plots of specimen dimension versus temperature. Measured values of 680 and 825 $^{\circ}\text{C}$ compared favorably with calculated values (668 and 842 $^{\circ}\text{C}$) [8].

Cylindrical test specimens were sectioned near the position of the thermo-couple that had been welded to the surface prior to heat treatment. Specimens were mounted, polished, and etched using conventional metallographic techniques. After final polishing, specimens were etched with 2% nitric acid in ethanol (2% nital), rinsed, and dried. These specimens were observed via light microscopy.

Specimens for transmission electron microscopy were prepared by cutting thin wafers from the mid-position of the specimen in close proximity to the attached thermocouples. The thin wafer was ground to about 0.1 mm in thickness. Discs of 3 mm in diameter were ground to a thickness of approximately 0.08 mm. Specimens were electro-polished in a jet polisher at 40 V using an electrolyte of CrO_3 , acetic acid, and water. Transmission electron microscopy was conducted at 120 kV.

Bright-field (BF) and centered-dark-field (CDF) imaging techniques were employed during transmission electron microscopy investigations. Additionally, critical information was obtained from electron-diffraction patterns, a common technique that has not been applied in sufficient detail to the microstructures observed. At 120 kV, the calculated electron wavelength is 0.00335 nm, and the nominal camera length used was 940 mm. A pure aluminum standard was used to determine a calibrated camera constant of $3.26 \times 10^{-12} \text{ m}^2$ (i.e., 32.6 $\text{mm} \cdot \text{\AA}$). Based on numerous analyses of electron-diffraction patterns from the microscope used in this study and the commonly selected operation parameters, the following uncertainties have been estimated: interplanar spacing $\pm 0.003 \text{ nm}$, e.g.,

$0.180 \pm 0.003 \text{ nm}$, interplanar angles $\pm 2^{\circ}$, e.g., $71^{\circ} \pm 2^{\circ}$, and $\pm 5^{\circ}$ for orientation relationships (ORs) or axis/angle pairs, e.g., $[1\bar{1}0]/55^{\circ} \pm 5^{\circ}$.

For results presented in this article, if the uncertainties listed above were exceeded, the data were considered to be of a dubious nature, and alternate interpretations were pursued. In some cases, satisfactory answers within these uncertainty ranges were not obtained, and in those cases, the analysis of those results is not provided.

Results and Discussion

CCT Diagram, Hardness, and Light Microscopy

Previous work [1] on 0.06% C HSLA-100 steel showed that the microstructure of a mill-processed plate that had been reaustenitized and quenched was predominantly martensitic, based on light-microscope images. Also, the microhardness (Vickers hardness, HV) of about 365 HV was consistent with as-quenched martensite for a carbon content of 0.06% [9]. Further evidence of a predominantly low-carbon martensitic microstructure was obtained from a CCT diagram [1], and Fig. 1 provides a slightly revised version of this diagram.

This diagram includes 14 different cooling curves, and each curve includes at least two points that reveal the start and completion of austenite transformation. These points were determined from computer-generated plots of specimen dilation as a function of temperature. For eight of these curves, numbers at the bottom of the figure indicate the measured microhardness values (HV) obtained from the as-cooled specimens. Additionally, to assist the interpretation of the transformation characteristics of austenite, Fig. 1 includes the labels A (austenite), M (martensite), AF (acicular ferrite), GF (granular ferrite), and PF (polygonal ferrite). Figure 2 shows microhardness as a function of the time interval associated with cooling from 800 to 500 $^{\circ}\text{C}$. In comparison with an “intermediate” A710-type composition [5], 0.06% C HSLA-100 steel shows a much more gradual decrease in hardness as cooling rate decreases and, concurrently, as martensite “transitions” to acicular ferrite with M/A constituent.

Light micrographs from specimens cooled at approximately 100 and 1 $^{\circ}\text{C/s}$ (i.e., $\Delta t_{800 \rightarrow 500}$ values of 3 and 315 s, respectively) are shown in Fig. 3. The dominant microconstituents are martensite at the higher cooling rate (100 $^{\circ}\text{C/s}$) and acicular ferrite with M/A at the lower rate (1 $^{\circ}\text{C/s}$). In Fig. 3(b), the features that are most-readily evident are small islands or pools of martensite and/or austenite (the M/A constituent). These features are particularly evident because the interfaces between crystals of acicular ferrite and pools of medium-carbon M/A are very

Fig. 2 Hardness versus time interval to cool from 800 to 500 °C

effective sites for etching with nital. However, crystals of acicular ferrite adjacent to one another frequently have the same crystallographic orientation, and so these adjacent crystals etch at a similar rate. Even in cases where there is a significant crystallographic misorientation between ferrite crystals, the etching effect is weak as compared with acicular–ferrite–M/A interfaces. Features labeled A, B, and C in Fig. 3(b) show different morphologies of M/A islands, where the island morphology is determined by the growth characteristics of the adjacent crystals of acicular ferrite. In comparison, Fig. 3(a) reveals no obvious medium-carbon M/A islands, consistent with the diffusionless formation of low-carbon martensite. For this microstructure, martensite packet boundaries appear to be the sites readily revealed by etching with nital. Two easily discernible adjacent packets of martensite are indicated at A and B in this figure. These same etching effects have been described in a previous paper concerning a similar steel of lower alloy content [5].

Despite the clear differences in the microstructures revealed by Fig. 3, many features are very small and worthy of more-detailed examination via transmission electron microscopy. Additionally, electron diffraction provides key crystallographic information that cannot be revealed by light microscopy. Acicular ferrite with M/A has been examined via transmission electron microscopy for a low-carbon copper-containing alloy of leaner composition than that of HSLA-100, and the reader is referred to that paper as being typical of the microstructure shown in Fig. 3(b) [5]. However, the microstructure in Fig. 3(a) for a low-carbon copper-containing alloy has been documented in much less detail. In a recent paper [9], transmission electron microscopy was used to examine a mill-processed 0.06% C HSLA-100 steel that had been re-austenitized and quenched. In the as-received condition,

Fig. 3 **a** Martensite (100 °C/s) and **b** acicular ferrite with M/A constituent (1 °C/s). Light micrographs, 2 pct. nital etch

its hardness was approximately 365 HV. The data from Figs. 1 and 2 show that a specimen with similar hardness (362 HV) was associated with a $\Delta t_{800 \rightarrow 500}$ value of 18 s, and thus a cooling rate calculated at 17 °C/s. This information was used to predict that the re-austenitized-and-quenched plate had been cooled in the mill at about 15 °C/s, and it was also used to select the laboratory specimen cooled at 17 °C/s for detailed transmission electron microscopy examination. This cooling rate is in the “middle” of the martensitic “range” and the associated microstructure is believed to be generally representative of that obtained at all the cooling rates within this range (labeled M in Fig. 1).

Transmission Electron Microscopy

An important aspect of this article is to present heretofore unpublished transmission electron microscopy results from laboratory processed 0.06% C HSLA-100 steel. Figure 4 shows a typical transmission electron microscope (TEM)

image of martensite formed during cooling at a rate of 17 °C/s. The individual crystals of martensite possess a lath-like morphology [12], and the apparent lath width (in this particular region at this tilt condition) is approximately 0.1 μm . The crystals of martensite show a moderate-to-high dislocation density in the as-cooled martensite structure and a dislocation density of approximately 10^{15} m^{-2} (i.e., 10^{11} cm^{-2}) is a reasonable estimate [13, 14]. This observation is consistent with volume accommodation effects during martensite formation [15, 16].

The intertwined nature of the laths in Fig. 4 is consistent with two or more variants of, for example, the Kurdjumov–Sachs OR [17] between the parent austenite phase and the product martensite phase. An austenite grain-boundary appears to have been present (prior to martensite formation) along the slightly curved linear feature that extends from A at the top of the micrograph to A at the bottom of the micrograph.

Figure 5(a) is a low-magnification BF TEM image of martensite laths, most of which are contained within a single prior austenite grain. Figure 5(b) is a CDF image of the same region produced from a (002) austenite reflection. It appears that the crystal of austenite in the upper left portion of Fig 5(b) also shows interlath retained austenite, apparently because this particular spot was common to both crystals of austenite. This statement does not imply that the two austenite grains had the same crystallographic orientation, and in fact would be an unlikely scenario for equiaxed adjacent grains of austenite formed during a simple re-austenitization step.

At higher magnification, individual laths of martensite appeared to be wholly “contained” within a single prior

Fig. 4 Lath martensite; bright-field TEM micrograph. A prior austenite grain-boundary appears to extend from A at the top to A at the bottom

grain of austenite, as expected [18]. Consistent with this comment, the arrows near the bottom of Fig. 5(b) show a continuous portion of retained austenite that delineates the boundary between this grain of austenite and the one below it. Similarly, arrows at the right side show another slightly less continuous grain-boundary “film.” These features would be uncommon or absent altogether if laths of martensite could grow from one grain into another of random crystallographic misorientation.

Figure 6 shows the selected-area electron-diffraction pattern from the central portion of the region shown in Fig. 5. At least four zones of martensite have been identified; however, only two zones have been indexed in Fig. 6(b). The magnitudes of the associated $\mathbf{g}_{hkl}$ vectors and the angular relationships between these vectors are consistent with a body-centered-cubic (BCC) phase with a

Fig. 5 Martensite laths within a single austenite grain. TEM micrographs: **a** BF image and **b** CDF image of retained austenite

Fig. 8 SADP of the central portion of the microstructure shown in Fig. 7. **a** Original pattern and **b** facsimile of this pattern with indexed spots from one zone of martensite (grid of *solid lines* and *numbers* without *highlights*) and one zone of spots from austenite (grid of *dashed lines* with *numbers* in *gray ovals*), and **c** a second zone of martensite. Electron beam direction = $[2\bar{1}0]$ for the first martensite zone, beam direction = $[\bar{5}31]$ for the second martensite zone, and beam direction = $[\bar{4}\bar{1}\bar{1}]$ for the austenite zone

presented first because the shape of this prior austenite grain appears to be easily envisioned. Excluding the illuminated regions of austenite in the upper left corner of Fig. 5(b), the equiaxed prior austenite grain in the center of this figure has a maximum chord length of about eight micrometers.

Figure 7 shows a second BF/CDF pair of micrographs revealing martensite contained within what again appears to have been another single grain of austenite; although, its apparent shape in two dimensions seems less “conventional” than that of Fig. 5(b). Nonetheless, its largest chord length in this field of view was measured to be about 7 μm . While this pair of micrographs is in some ways less revealing than those of Fig. 5, the associated electron-diffraction pattern was interpreted in a more-complete fashion than that of Fig. 6.

Figure 8 shows an SADP from the central region of Fig. 7. Specifically, Fig. 8(a) is the original pattern, (b) is a facsimile of the pattern that includes labels for indexed zones of martensite spots and austenite spots, and (c) is another facsimile with a second indexed zone of martensite spots. From Fig. 8, the magnitudes of the associated $\mathbf{g}_{hkl}$ vectors and the angular relationships between these vectors are consistent with a BCC phase with a lattice parameter of approximately 0.287 nm and a face-centered-cubic (FCC) phase with a lattice parameter of about 0.360 nm, i.e., consistent with values expected for martensite and austenite. Detailed analysis of this pattern revealed an OR between martensite (M) and austenite (A) phases that was within 5° of

$$(\bar{1}10)_M // (\bar{1}11)_A$$

$$(\bar{1}\bar{1}\bar{1})_M // (\bar{1}0\bar{1})_A$$

$$(\bar{1}\bar{1}2)_M // (\bar{1}\bar{2}1)_A$$

and also within 5° of

$$(001)_M // (0\bar{1}1)_A$$

$$(\bar{1}10)_M // (\bar{1}11)_A$$

$$(\bar{1}\bar{1}0)_M // (\bar{2}\bar{1}\bar{1})_A$$

The first relationship is a variant of the Kurdjumov–Sachs OR [17], while the second is a nearby variant of the Nishiyama–Wassermann OR [19, 20]. These ORs are related by a 5.26° rotation about the common parallel axes

Fig. 9 Martensite laths within a single austenite grain. TEM micrographs: **a** BF image and **b** CDF image of retained austenite

of $(\bar{1}10)_M // (\bar{1}11)_A$. Based on the estimated uncertainty, it is not possible to determine which OR fit the experimental data the closest. As the main goals of this study are to characterize key microstructural features via imaging and to identify important crystallographic information via electron diffraction, the quoted accuracy is sufficient to prove the point. Specifically, the phenomenological theory of martensite crystallography requires a reproducible OR

Fig. 10 SADP of the central portion of the microstructure shown in Fig. 9. **a** Original pattern and **b** facsimile of this pattern with indexed spots from one zone of martensite (grid of solid lines and numbers without highlights) and two spots from austenite (black numbers in white ovals), and **c** a second zone of martensite. Electron beam direction = $[111]$ for the first martensite zone, and beam direction = $[001]$ for the second martensite zone

between the parent and the product phases [18], and those reported by Kurdjumov–Sachs and Nishiyama–Wassermann [17, 19, 20] are most often observed for carbon steel.

A final example of martensite laths formed within a single grain of austenite is shown in Fig. 9. The amount of retained austenite shown in Figs. 5(b), 7(b), and 9(b) is similar to previous work [9], and a conservative estimate is about 5 vol.%. Measurement of the amount of retained austenite via x-ray diffraction for this steel was hampered by the low intensity, owing to the small amount of austenite, and peak broadening, owing to the small size of the interlath films. Thus, the amount of austenite in the microstructure was underestimated by x-ray techniques and is not reported. Additionally, a maximum chord length from this grain of austenite was measured at about 9 μm .

The associated electron-diffraction pattern is shown in Fig. 10. Facsimiles of the original pattern reveal two zones of martensite, but only a single row of spots from the austenite phase was identified with certainty (similar to Fig. 6). A key aspect of the martensite patterns was the axis/angle pair of approximately $[1\bar{1}0]/55^\circ$. This axis angle pair is close to two different variant pairs of the Kurdjumov–Sachs OR. This and other variant pairs have been examined in detail Morito et al. [21, 22], and the variant pair observed here is consistent with the minimization of the overall shape strain for martensite crystals formed in a grain of austenite.

In summary, the complexity of electron diffraction information presented in this study was such that no single example (i.e., Figs. 6, 8, 10) was identified that could reveal all the key diffraction information at once. However, the three examples collectively reveal data that are consistent with (i) the expected phases (martensite and austenite), (ii) a frequently reported OR for these phases, and (iii) variant pairing that permits minimization of the overall shape strain associated with two variants of martensite formed in the same grain of austenite.

So far in this article, evidence has been presented that is consistent with low-carbon lath martensite microstructure with about 5% of retained austenite. Other, less-frequently observed features will be described next. Although the majority of the martensite laths had moderate-to-high dislocation densities, best shown by Fig. 1, some laths contained twinned regions. Twins in martensite are evidence of strain accommodation during martensite formation from austenite and are not observed within crystals of acicular ferrite.

Figure 11 shows an example of twins within a lath of martensite in both BF and dark-field conditions. The associated selected-area electron-diffraction pattern from this region, Fig. 12, shows two zones of martensite spots

Fig. 11 Twins within crystals of lath martensite. TEM micrographs: **a** BF image and **b** CDF image of twins. Twins at A and B seem to be rather coarse, while those at C are very fine

that reveal the twin relationship between BCC crystals. In the original pattern, numerous double-diffraction spots are evident, and the arrows point to several such spots. The coarse nature of the twins at A in Fig. 11 are similar to those reported previously for this steel [9]. Finer, but more-difficult-to-see twins are evident at C.

An additional fine feature occupying a small portion of the microstructure is revealed by Fig. 13. In Fig. 13(a), dark “platelets” of a second phase are revealed, and the associated selected-area electron-diffraction pattern is provided in Fig. 13(b). The rotation between the two images has been accounted for in the placement of these figures. Diffraction spots from these dark regions were not obtained; however, the shapes of these features and the approximate habit planes of $\{101\}$ with respect to the martensite strongly suggest the presence of cementite, as observed elsewhere [23]. This type of multi-variant cementite precipitation is characteristic of tempered martensite, and in this case it formed during the quench.

Owing to the low-carbon level of this steel, the volume fraction of cementite is rather low, and combined with

Fig. 12 SADP from the region near A in Fig. 11. **a** Original pattern and **b** facsimile of this pattern with indexed spots from the martensite “matrix” (M) indicated by solid black lines and numbers, and spots from the twins (T) with dashed black lines with black numbers in white ovals. Beam direction = $[1\bar{1}3]_M/[3\bar{1}1]_T$

different variants of the cementite, it is not entirely surprising that electron-diffraction spots from this phase are not evident. These features and other precipitates make up <1% of the as-cooled microstructure for this steel. The cementite platelets were typically observed in the largest martensite laths. There was little if any evidence of other carbide phases or copper-rich precipitates; however, the intent of this study was to document the base microstructure and its key features, and not to examine fine-scale precipitation. It is suggested that these cementite precipitates are formed during autotempering, and that they are observed in the largest or first-formed martensite laths that form nearest to the M_s temperature upon cooling.

Fig. 13 At least three variants of autotempered cementite precipitates within a large crystal of lath martensite. **a** Bright-field TEM image and **b** indexed EDP from the martensite crystal. Beam direction = $[1\bar{1}1]$. The image rotation between these images has been accounted for in their placement above

Finally, while the large majority of the regions investigated via thin-foil transmission electron microscopy displayed characteristics consistent with a martensitic microstructure, a very limited amount of acicular ferrite appeared to be present, although evidence will not be presented here. The difference between martensite and acicular ferrite was most-readily revealed by the presence of “blocky” islands of retained austenite between adjacent crystals of acicular ferrite as compared with interlath thin films of austenite between laths of martensite. These differences are being investigated further, but as of this time, the differences have not been documented in sufficient detail.

Summary and Conclusions

A CCT diagram from a steel with chemical composition (in wt.%) of: 0.06 C, 0.83 Mn, 0.010 P, 0.002 S, 1.66 Cu, 3.48 Ni, 0.58 Cr, 0.59 Mo, 0.37 Si, 0.028 Nb, 0.028 Al, balance Fe was reexamined and, in combination with hardness data and light-microscope images, the microstructure observed over a wide range of cooling rates was martensitic. A laboratory processed specimen cooled at approximately 17 °C/s was chosen for detailed examination via transmission electron microscopy.

Conventional BF images were complemented with CDF images and electron diffraction to reveal microstructural and crystallographic features that are consistent with low-carbon lath martensite. These features include crystals of a lath-like nature, with a moderate-to-high dislocation content. Crystals of martensite were contained within single prior austenite grains, and occasionally regions of twinned martensite were observed. About 5 vol.% of interlath retained austenite was present. The martensite/austenite OR was close to that reported by Kurdjumov–Sachs. Adjacent regions of martensite possessed two different crystallographic variants of the Kurdjumov–Sachs OR. Some of the largest laths were autotempered, displaying multiple variants of cementite platelets.

These results are consistent with previous work on the same steel that had been mill processed with a final re-austenitization-and-quench step. In both cases, the hardness was about 360 HV and the prior austenite grain structure was equiaxed with a grain size of about 10 µm.

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