Quenched and Partitioned Microstructures Produced *via* Gleeble Simulations of Hot-Strip Mill Cooling Practices

G.A. THOMAS, J.G. SPEER, and D.K. MATLOCK

Previous researchers reported on quenched and partitioned (Q&P) microstructures produced *via* carbon partitioning from martensite into austenite during isothermal annealing after quenching to develop a partially martensitic initial structure. However, the thermal profile used in previous studies is not well suited to creating Q&P microstructures directly from a hot-strip mill. In this work, the commonly employed Q&P thermal profile (*i.e.*, having an isothermal partitioning step) was modified to evaluate nonisothermal partitioning that might instead occur during cooling of a wound coil. Thus, it was possible to assess the potential for creating Q&P microstructures and properties directly off of the hot mill. Gleeble thermal simulations representative of a hot-strip mill cooling practice were used to create dual-phase, Q&P, transformation-induced plasticity (TRIP), and conventional microstructures by varying the quench/coiling temperatures (CTs) using a 0.19C-1.59Mn-1.63Si (wt pct) steel. Microstructural and mechanical property data indicate that hot rolling might be a viable processing route for high-strength Q&P steels.

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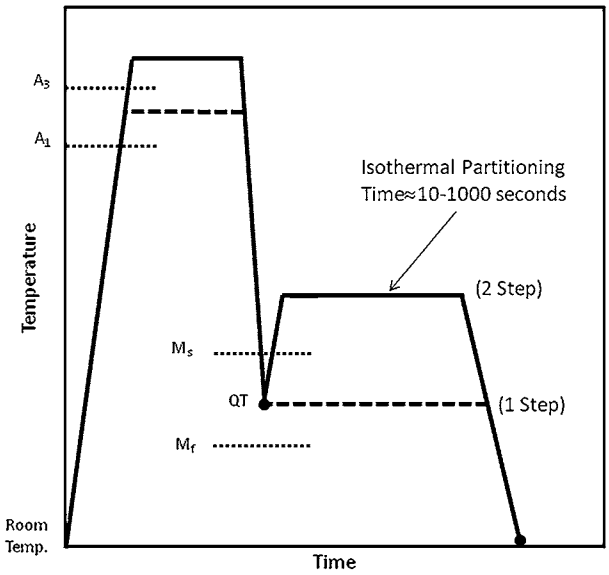
I. INTRODUCTION

QUENCHED and partitioned (Q&P) steels exhibit excellent combinations of strength and ductility, because they contain a significant fraction of martensite along with austenite that can transform to martensite during deformation leading to enhanced work hardening and improved elongation.[1–6] Previous researchers created Q&P microstructures *via* thermal profiles that are perhaps most suited to an annealing step following cold rolling, as shown schematically in Figure 1(a). In these profiles, the steel is austenitized (either fully or partially) and then quenched to a temperature between the martensite start (M_s) and martensite finish (M_f) temperatures to create a controlled mixture of martensite and austenite (and any intercritical ferrite present during annealing). The steel is then held isothermally at the quench temperature (QT) (one-step partitioning) or at an elevated temperature (two-step partitioning) to allow carbon to partition into austenite from martensite. Carbon-enriched austenite, stable at room temperature, is capable of contributing to mechanical properties in the same manner as it does in transformation-induced plasticity (TRIP) steel. Other microstructural changes due to conventional tempering reactions (*e.g.*, carbide formation or carbon atom trapping at defects in the martensite), decomposition of austenite into other transformation products such as bainite, or interface migration are recognized as potential competing mechanisms during the partitioning step.

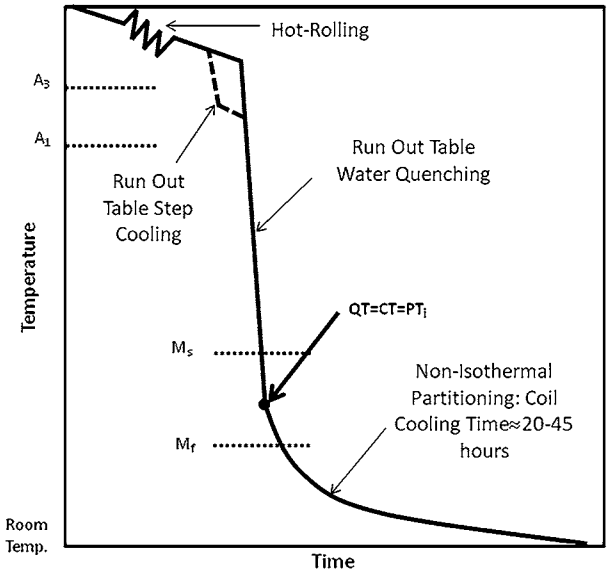
The present authors previously proposed a processing route that would allow for the production of Q&P microstructures directly off of the hot-strip mill,[7] as shown schematically in Figure 1(b). Here, the quenching step is accomplished during run-out table cooling, similar to that in dual-phase steels (step cooling may be used prior to quenching if primary ferrite is desired). Further, the partitioning step may be accomplished nonisothermally during slow cooling of the wound coil, similar to that in austempering of hot-rolled TRIP steel. Under these conditions, the coiling temperature (CT) dictates critical attributes of the process. First, the coiling temperature serves as the QT, which sets the initial fraction of martensite and austenite prior to partitioning. Second, the coiling temperature serves as the initial partitioning temperature (PT_i) and ultimately establishes the extent of partitioning that can occur during coil cooling. Because these two important parameters are both controlled by the coiling temperature, the hot-rolled Q&P process is fundamentally more constrained than the profile shown in Figure 1(a) in which the QT and partitioning temperature (and time) can be independently controlled.

Figure 2 shows the calculated characteristic diffusion distances, x_c, of carbon through (a) austenite or (b) martensite for nonisothermal temperatures associated with coil cooling from various coiling temperatures compared with those of isothermal partitioning conditions used by previous Q&P researchers. The details of these calculations are discussed elsewhere,[7] and the conclusion is that the heat retention during coil cooling may provide a suitable partitioning response for coiling temperatures that might also serve as suitable QTs (*e.g.*, ~523 K (250 °C)[2]). The gray bands in Figure 2

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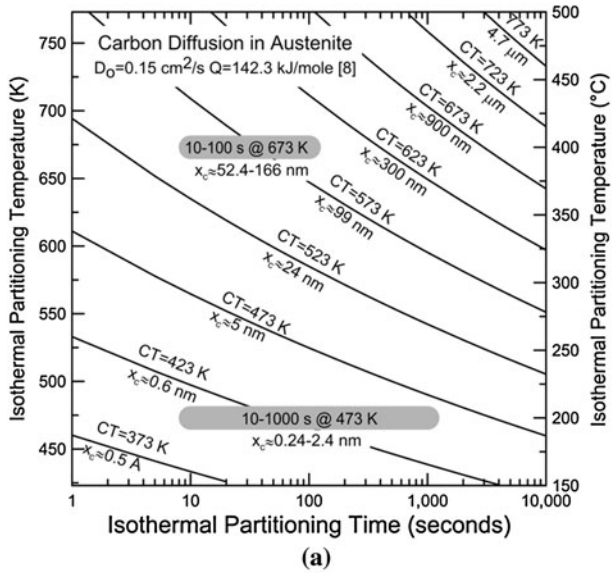


(a)

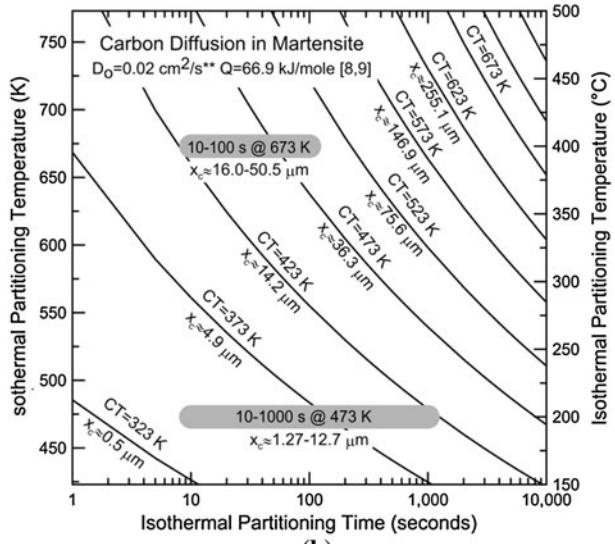


(b)

Fig. 1—(*a*) Schematic thermal profile of the Q&P process used by previous researchers, involving an isothermal partitioning step.[1–6] (*b*) Schematic thermal-mechanical profile proposed to create Q&P microstructures directly from the hot-strip mill. PT_i designates the initial partitioning temperature dictated by the CT, which is also the QT.[7]



(a)



(b)

Fig. 2—Locus of nonisothermal partitioning responses during coil cooling from CTs that are equivalent to isothermal partitioning at temperatures and times indicated by the coordinate axes. Modified from Ref. 7. Isopleths represent CTs, which yield characteristic diffusion distances, x_c, indicated on the figure. The calculations are based on carbon diffusion in (*a*) austenite[8] or (*b*) martensite.[9,10] The shaded bands indicate examples of isothermal partitioning conditions used in earlier studies,[3,4] at 473 K and 673 K (200 °C and 400 °C), along with associated characteristic carbon diffusion distances. **The source reporting the activation energy for carbon diffusion in martensite does not report a value of D_o;[10] thus, D_o is taken as that of carbon diffusion in ferrite.[9]

represent isothermal partitioning conditions (along with associated diffusion distances) used by previous Q&P investigators that yielded significant fractions of retained austenite.[3,4] As shown in Figure 2, the characteristic carbon diffusion distances through austenite or martensite associated with cooling from coiling temperatures near the optimum QT for Q&P[2] are similar to (or greater than, in the case of carbon diffusion through martensite) those for isothermal partitioning conditions performed by previous researchers. Thus, Figure 2 suggests that the heat retention associated with coil cooling is more than sufficient for carbon to escape from martensite and similar to that in previous Q&P research for homogenization of carbon in austenite. Therefore, it is conceptually possible to produce Q&P microstructures directly from the hot-strip mill, and the present work presents the experimental results of a testing matrix designed to validate the calculations. The experimental matrix varied the coiling temperature to create dual-phase, Q&P, TRIP, and ferrite-pearlite microstructures in a single steel alloy by thermal processing representative of hot-strip mill cooling practices. Microstructures

and mechanical properties were evaluated and compared to those of previous Q&P literature, which used isothermal partitioning conditions.

II. EXPERIMENTAL PROCEDURE

The steel used for this work was a 0.19C-1.59Mn-1.63Si (wt pct) commercially cold-rolled TRIP sheet steel. This composition was chosen because it allows for practical simulations of both TRIP and Q&P steels. The martensite start temperature calculated for this alloy is 671 K (398 °C).[11] If 25 pct intercritical ferrite and thus an intercritical austenite carbon level of 0.25 wt pct is assumed (as previously reported for the intercritical temperature used in this work[3,4]), then the M_s of the intercritical austenite is calculated to be 644 K (371 °C).[11] Thermal processing was conducted using a Gleeble 1500 (Dynamic Systems Inc., Poestenkill, NY) on sheet specimens with dimensions of 127 mm × 25.4 mm × 1.2 mm. The thermal profiles used for Gleeble simulations are shown schematically in Figure 3(a). The samples were heated to the austenitizing (1173 K (900 °C)) or intercritical temperature (1093 K (820 °C)) in 60 seconds and held 180 seconds at the austenitizing or intercritical temperature. Deformation was not included in the Gleeble processing, because the constraints on required specimen geometry would not allow for subsequent tensile testing. Nonetheless, the aforementioned annealing steps were anticipated to create microstructures expected after hot rolling and recrystallization (and step cooling to form primary ferrite in the case of the intercritically annealed samples). Following austenitization, run-out table water cooling was simulated by helium quenching. Simulated coiling temperatures ranged from 373 K to 773 K (100 °C to 500 °C) for intercritically annealed samples and 473 K to 573 K (200 °C to 300 °C) for fully austenitized samples. In both cases, the coiling temperature was varied in steps of 25 K (25 °C) over the range indicated. Additionally, samples were quenched directly to room temperature for both the intercritically annealed and fully austenitized conditions. Samples that were intercritically annealed (at 1093 K (820 °C)) prior to helium quenching are designated "IC," and those that were fully austenitized (at 1173 K (900 °C)) prior to helium quenching are designated "FA." In both cases, the quench/coiling temperature (in Kelvin) follows the letter designation.

Figure 3(b) shows a thermal profile measured during helium quenching to room temperature. All simulations followed the same thermal profile during quenching to the appropriate QTs. A comparison of the observed quench rates with time-temperature-transformation (TTT) diagrams for similar steels indicates that at the imposed quench rates, some nonmartensitic transformation products such as bainite may have formed.[12] While bainite formation complicates the understanding of mechanisms responsible for carbon enrichment of austenite, the cooling rates achieved here may be similar to those in an industrial hot-strip mill and should thus provide a meaningful simulation of industrial processing.

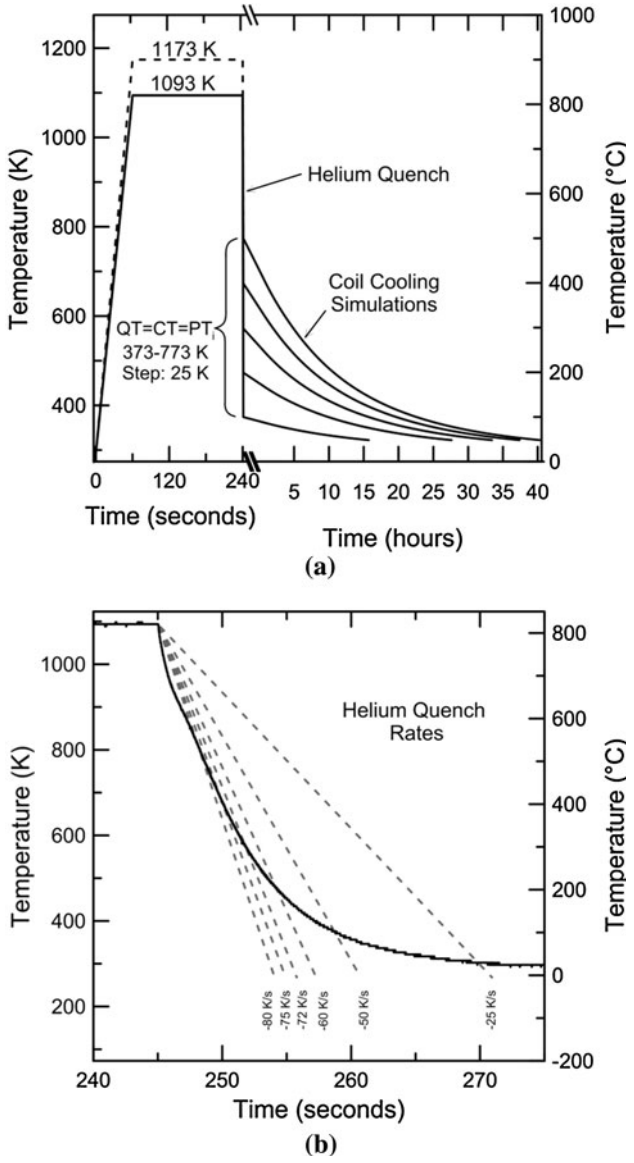


Fig. 3—(*a*) Thermal profiles used for Gleeble simulations of hot-strip mill cooling practices. The helium quenching rate is not depicted accurately. (*b*) Measured thermal profile during helium quenching to room temperature (solid line). All of the simulations followed the same thermal profile during quenching to the appropriate QT. The dashed lines represent the average cooling rates to QTs of 773 K, 673 K, 573 K, 473 K, 373 K, and 293 K (500 °C, 400 °C, 300 °C, 200 °C, 100 °C, and 20 °C).

After helium quenching, the samples were subjected to simulated coil cooling from the quench (coiling) temperature to room temperature. All of the coiling simulations were programmed to follow the same cooling profile (shown in Figure 3(a)),[7,13] albeit starting at different points along the coil cooling profile. Pretrials indicated that the surface temperature was uniform within about ±3 K (±3 °C) over the area of interest during the Gleeble simulations.[14]

Retained austenite volume fractions and austenite carbon contents were estimated *via* X-ray diffraction (XRD). Samples were prepared by lightly sanding to remove surface oxides, and then chemically thinning for

approximately 3 minutes in a water-cooled container of 10 parts water: 10 parts 30 pct hydrogen peroxide: 1 part hydrofluoric acid. Thinning reduced the sample thickness by about 10 μm. XRD was performed with nickel-filtered copper radiation and a PHILIPS* X-pert diffractometer operating at 45 kV and 40 mA, equipped with an X'celerator detector and a 1 deg slit. Four austenite and four ferrite/martensite peaks were quantified by scanning through a 2-theta range from 40 to 105 deg over a total scan time of about 21 minutes. The volume fraction of retained austenite was calculated from the integrated peak intensities and positions according to the SAE method.[15] The average austenite carbon content was calculated based on expansion of the measured lattice parameter.[16]

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Metallographic samples were polished and etched with 2 pct nital. All samples were viewed in the longitudinal through-thickness plane. Scanning electron microscopy (SEM) evaluations were performed using a field emission SEM operating at 5 kV with a working distance of 10 mm. Electron backscattered diffraction (EBSD) analyses were performed on selected samples to confirm the morphology and location of retained austenite. Samples were prepared for EBSD by polishing with 6-, 3-, 1-, and 0.05-μm diamond slurries. The final polishing step was performed on a vibratory polisher using 0.05-μm colloidal silica for approximately 18 hours. The accelerating voltage for EBSD data collection was 20 kV, and the working distance was about 15 mm for all EBSD analyses. The step size for the EBSD data collections ranged from 10 to 50 nm using a hexagonal grid pattern.

Subsized tensile specimens with reduced gage lengths of 12 by 5 mm were machined from the Gleeble samples; the complete specimen dimensions were published previously.[7] Uniaxial tensile tests were performed at a constant crosshead speed of 2.54 mm/min. Displacement within the reduced gage of the subsized tensile samples was measured using an 8-mm extensometer. The maximum extensometer strain range was 15 pct, which was less than the total strain of some of the samples. Therefore, tests were paused at 14 pct strain to reset the extensometer without removing the load. The tensile test was then resumed and data were corrected for the new gage length and added to the original displacement data to create a continuous set of load-displacement data. Prior to using the nonstandard subsized tensile sample for this current study, the validity of data from the subsized samples was confirmed with a series of experiments on a commercially produced DP600 (dual-phase) steel. Standard ASTM E-8 sheet tensile samples (50.8-mm gage length by 12.7-mm width) and 12-mm gage length subsized tensile samples were machined with the same orientation with respect to the rolling direction. Four samples of each orientation were tested and the resulting stress strain data were shown to be identical up to the maximum load, *i.e.*, the onset of necking. As anticipated, beyond necking, measured strain data on the subsized samples were significantly higher than observed on the standard sample. Thus, in this article, only mechanical property data obtained prior to necking are considered, as these data were shown to be independent of sample size.

III. RESULTS AND DISCUSSION

A. *Microstructure*

The austenite volume fractions and average austenite carbon contents of the samples subjected to Gleeble simulations are shown in Figure 4. For comparison, Figure 4(a) also shows the austenite volume fractions reported by Clarke[3] and Streicher *et al.*[4] as well as the weight fraction of austenite calculated by the method proposed by Speer *et al.*[2] assuming "ideal" full partitioning. As seen in the figure, the austenite fractions measured for the intercritically annealed samples (IC samples) are similar to or greater than those reported by Clarke. Further, the final austenite fraction seems less sensitive to coiling temperature than calculated. This insensitivity to coiling temperature may favor the ability to commercially produce hot-rolled Q&P in mills in which accurate coiling temperature control may be challenging in the temperature regime of interest. The austenite fractions of the fully austenitized samples (FA samples) are significantly less than those of the IC samples. However, austenite carbon contents of the IC and FA samples are significantly enhanced as compared to the bulk alloy carbon concentration; further, the carbon enrichment levels are similar to those reported by Clarke for Q&P and austempering treatments (generally, 1.0 to 1.4 wt pct C) in the same steel.[3,4]

Secondary electron micrographs of selected representative samples are shown in Figures 5 and 6. As shown in Figure 5(a), the intercritically annealed samples (IC samples) coiled between 293 K and 448 K (20 °C and 175 °C) generally exhibit microstructures typical of dual-phase steels, *i.e.*, martensite and intercritical ferrite. However, in most cases, bainitic ferrite (or perhaps Widmanstätten ferrite) was observed, which may be the result of insufficient cooling rates during helium quenching. Further, as reported in Figure 4, most of the retained austenite fractions are significantly higher than those typical of dual-phase steels.[17]

Samples coiled between about 448 K and 623 K (175 °C and 350 °C) exhibited microstructural features typical of those observed in previous Q&P literature, as shown in Figures 5(b) through (d) and at higher magnification in Figure 6(a), an enlargement of the boxed region in Figure 5(c). The characteristic features of Q&P steels are martensite blocks with austenite adjacent to martensite as pools or between martensitic laths as thick films, which were presumably stabilized by carbon diffusing from martensite into austenite during the partitioning step. Examples of these features are indicated with arrows in Figures 5(b) and 6(a). Some TRIP-like bainite features were also observed in these samples, for example, the circled region in Figure 5(b)

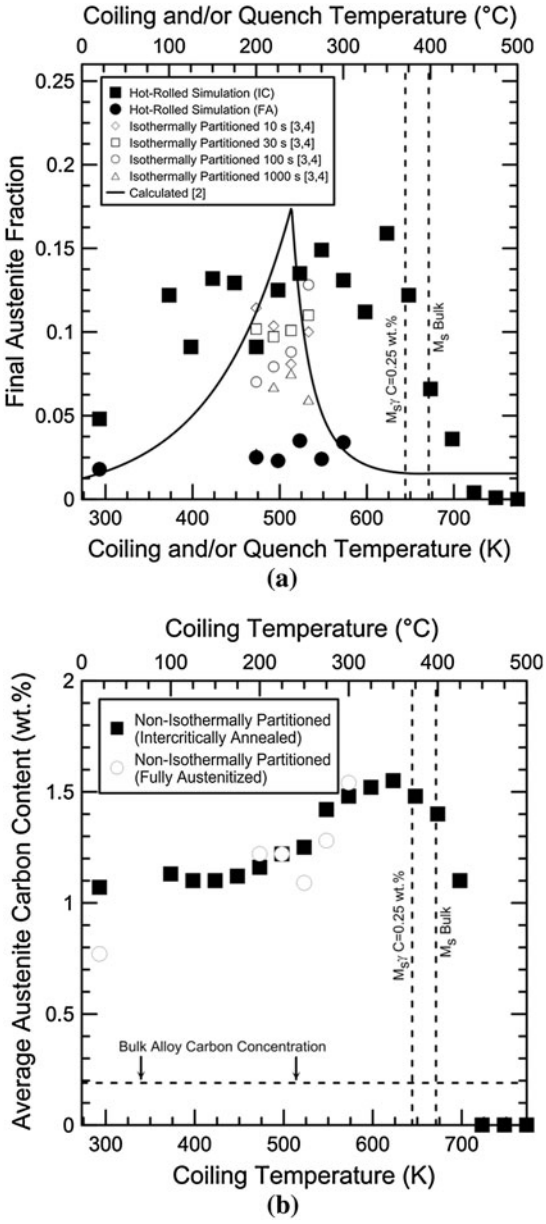


Fig. 4—(*a*) Austenite volume fractions for samples in this work and that of Clarke[3] and Streicher *et al.*[4] The solid curve represents the retained austenite weight fractions calculated[2] assuming ideal full partitioning and 0 or 25 pct intercritical ferrite. (*b*) Final austenite carbon contents for samples in this work. M_s temperatures calculated using the method of Mahieu *et al.*[11] are also shown for the bulk alloy composition as well as for intercritical austenite assuming 0.25 wt pct carbon.

exhibits regions of bainitic ferrite separated by elongated regions of retained austenite that were likely carbon enriched during bainite formation, the mechanism primarily responsible for austenite enrichment during austempering of TRIP steels. The extent to which the aforementioned mechanisms are responsible for carbon enrichment of austenite likely varies with coiling temperature. At lower coiling temperatures (~448 K to 523 K (175 °C to 250 °C)), where bainitic ferrite is not as prevalent, most of the austenite was likely stabilized by carbon partitioning from martensite; with increasing coiling temperature, and increasing fraction of bainitic ferrite, austempering likely contributes to a greater extent to carbon enrichment of austenite. This is especially true for samples with coiling temperatures near the M_s temperature of the intercritical austenite (~644 K (371 °C)[11]). For example, refer to Figure 5(e), where bainitic ferrite is prevalent throughout the microstructure and martensite regions are limited. Thus, samples with coiling temperatures near the M_s are considered primarily TRIP-like. EBSD analyses were performed on selected samples to confirm the morphology and location of retained austenite. Although not presented here, the results of the EBSD analysis confirmed that the regions identified as retained austenite in the preceding SEM analyses, in fact, were correctly identified.

The highest coiling temperatures (~723 K to 773 K (450 °C to 500 °C)) exhibited microstructures containing pearlite with very fine interlamellar spacing as well as grain boundary carbides, as shown in Figure 5(f). The fully austenitized samples were primarily martensitic with some retained austenite (2 to 4 pct as measured by XRD). Figure 6(b) shows the microstructure of one of the fully austenitized samples (sample FA-523). Note that some ferrite was also observed, interpreted to have formed on cooling, at the imposed helium quenching.

B. *Mechanical Properties*

Figure 7 shows the yield strength, ultimate tensile strength (UTS), and uniform elongation of the intercritically annealed samples (IC samples) as a function of coiling (quench) temperature. Figure 7 also indicates the approximate temperature ranges in which each of the various microstructure types were observed. The temperature ranges are approximate and there is overlap where mixed microstructures were present. The UTS is insensitive to coiling temperatures below approximately 498 K (225 °C) and above about 623 K (350 °C). The tensile strengths of the intermediate coiling temperatures (~498 K to 623 K (225 °C to 350 °C)) transition between two "plateaus" observed in the UTS values. The yield strength generally increases with increasing coiling temperature; therefore, the yield to tensile strength ratio increases with increasing coiling temperature. The uniform elongation increases slightly with coiling temperature up to about 573 K (300 °C) and then at a faster rate up to a peak at a coiling temperature of about 623 K (350 °C). The mechanical properties are sensitive to coiling temperature in the range of coiling temperatures that produce Q&P steels with enhanced elongation properties (*i.e.*, ~498 K to 623 K (225 °C to 350 °C)), and coiling temperature control would require attention in any efforts to commercialize this approach. The tensile properties of the fully austenitized samples (FA samples) were also measured. The FA samples exhibited tensile strengths between about 1100 and 1400 MPa with uniform elongations between about 4 and 7 pct; because the intercritically annealed samples exhibited greater uniform elongation values (and greater austenite fractions), the remainder of this article is focused on the intercritically annealed samples.

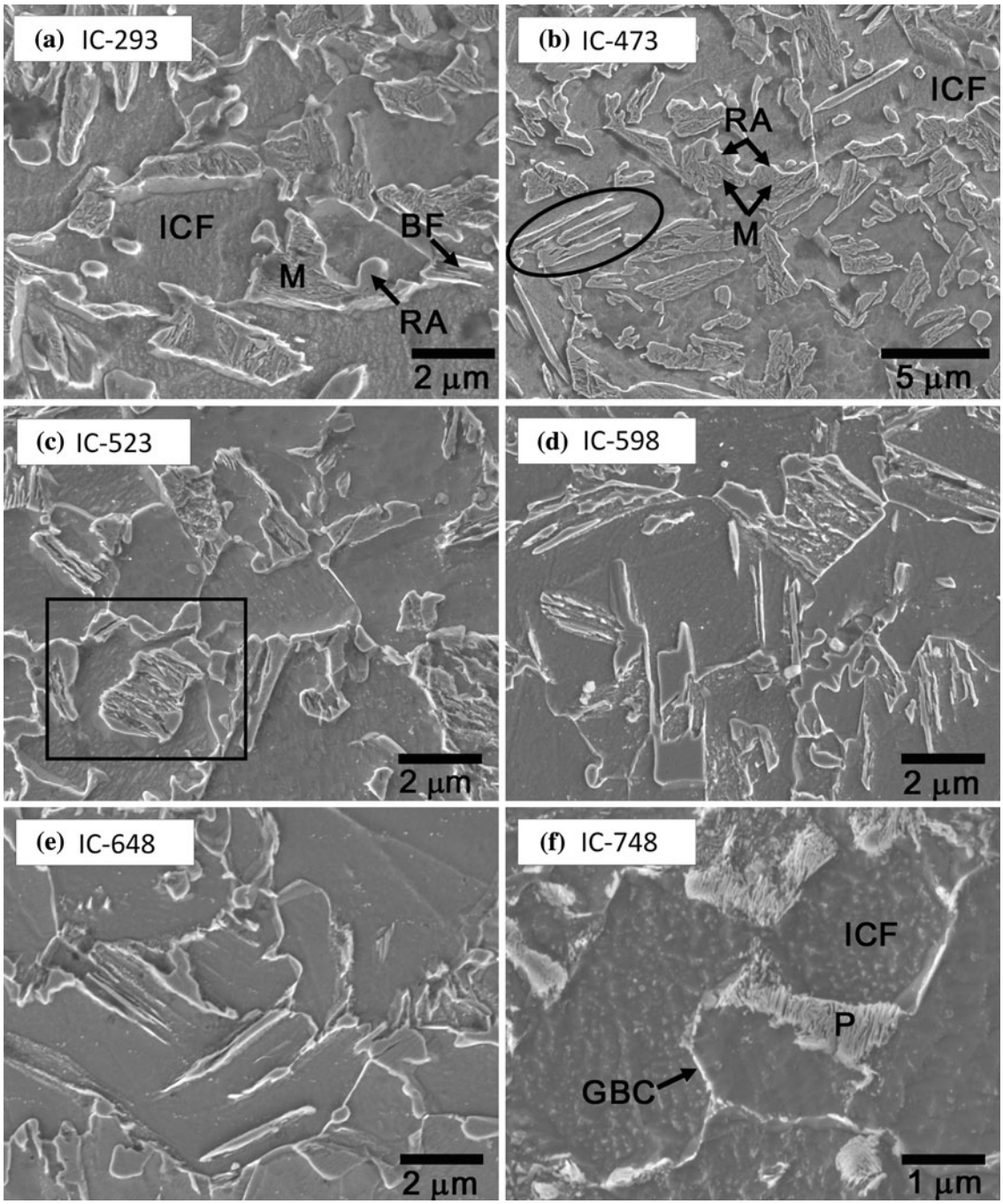


Fig. 5—Representative SEM images of the sample at various magnifications to highlight characteristic features. (*a*) IC-293, (*b*) IC-473—the circled region exhibits regions of bainitic ferrite separated by elongated regions of retained austenite, (*c*) IC-523—the boxed region exhibits features characteristic of Q&P and is enlarged in Fig. 6(a), (*d*) IC-598, (*e*) IC-648, and (*f*) IC-748. ICF, M, RA, BF, GBC, and P indicate intercritical ferrite, martensite, retained austenite, bainitic ferrite, grain boundary carbide, and pearlite, respectively. Etched with 2 pct nital.

The mechanical property trends in Figure 7 appear to correspond with the indicated microstructure variations. The steels exhibiting the dual-phase microstructures (*i.e.*, the lower coiling temperatures) exhibited the lowest yield strengths with the highest tensile strengths. This is interpreted to reflect a high density of mobile dislocations and the internal stresses caused by the shear and volume expansion associated with martensite formation from austenite. The unpinned dislocations around the martensite allow the steel to yield continuously. With increased coiling temperature, the extent of aging of the ferrite around the martensite presumably increases (*i.e.*, some dislocations become pinned by solute atoms and others are recovered). Thus, the yield strength increases slightly with increasing temperature; however, sufficient numbers of unpinned dislocations are present such that

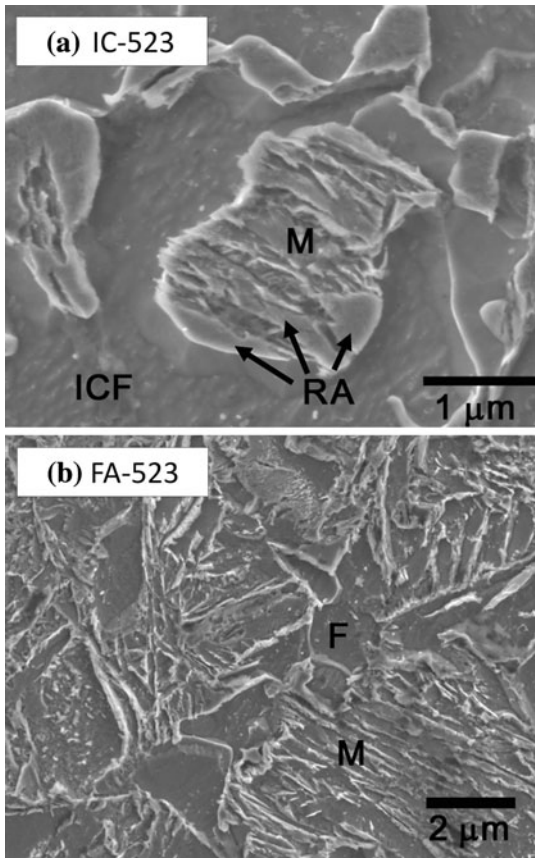


Fig. 6—SEM images of samples (*a*) IC-523 (enlarged from Fig. 5(c)) and (*b*) FA-523. ICF, M, and RA indicate intercritical ferrite, martensite, and retained austenite, respectively.

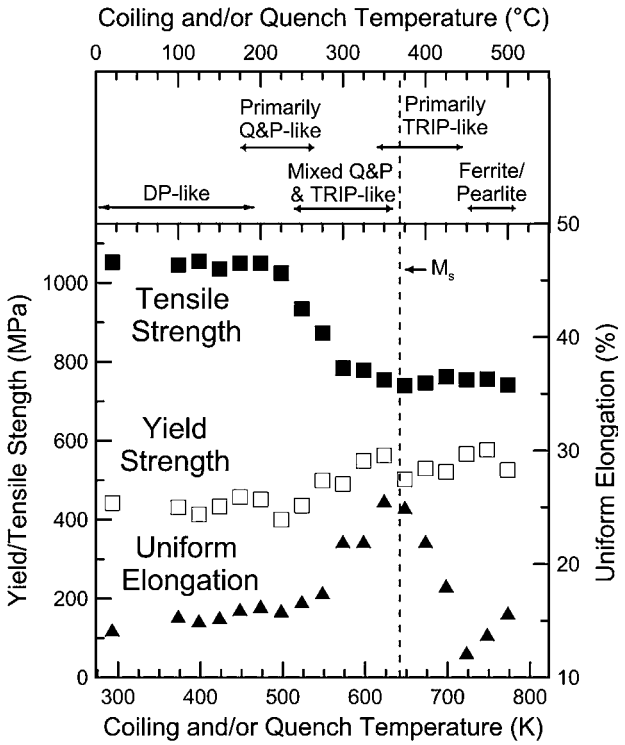


Fig. 7—Tensile properties as a function of coiling (quench) temperature for samples that were intercritically annealed and then quenched and subjected to a coil cooling profile (IC samples). UTSs and yield strengths are plotted on the axis to the left. Uniform elongations are plotted on the axis to the right. The CT ranges in which each of the types of microstructures were observed are indicated qualitatively above the plotted data. The indicated regions are approximate.

the steel exhibited continuous yielding behavior in all samples coiled below 573 K (300 °C).[14] In the coiling temperature range from ~448 K to 523 K (175 °C to 250 °C), microstructures were observed that had primarily Q&P-like character. The mechanical properties of the Q&P microstructures transition between those of dual-phase microstructures (high tensile strength with lower elongation) and the TRIP microstructures (lower tensile strength with higher elongation) in this range of coiling temperatures. The TRIP microstructures contained significant fractions of retained austenite stabilized *via* the formation of bainitic ferrite. The fraction of bainitic ferrite increased with increasing coiling temperature (up to the M_s). The samples coiled between 548 K and 698 K (275 °C and 425 °C) exhibited significantly higher uniform elongations, while the UTS remained relatively unchanged. This range of coiling temperatures included some samples that were classified primarily as Q&P microstructures and some that were classified as TRIP microstructures, with mixed microstructures at intermediate temperatures within this regime. These samples exhibited work hardening characteristics involving a constant or increasing instantaneous strain hardening exponent with true strain,[14] often associated with TRIP behavior.[3,6,18] The transformation from retained austenite to martensite increases uniform elongation by virtue of the volume expansion associated with the transformation, thus delaying the geometric instability condition, while maintaining high UTSs due to the formation of a harder phase (martensite), the generation of additional dislocations, and the resulting work hardening effect.

Figure 8(a) shows the uniform elongation as a function of the UTS for the samples in this work as well as those of Clarke[3] and Streicher *et al.*[4] using the same steel, as well as those reported by De Moor *et al.*[5,6] using a similar 0.2C-1.63Mn-1.63Si (wt pct) steel. As seen in the figure, many of the hot-rolled sheet simulations resulted in property combinations within the "envelope" of the cold-rolled sheet properties found by previous researchers using isothermal partitioning conditions. Further, despite the low austenite fractions observed in the fully austenitized samples (FA samples) here, the mechanical properties obtained in some cases compare favorably with those reported by De Moor for fully austenitized Q&P samples. Figure 8(b) is an expanded plot of Figure 8(a) showing only the results for hot-rolling simulations that involved intercritical annealing, labeled with their respective coiling temperatures and qualitative microstructural characterizations. Examination of Figures 8(a) and (b) indicates that the mechanical property combinations of the nonisothermally processed Q&P samples in this work were similar to those found by previous researchers using isothermal

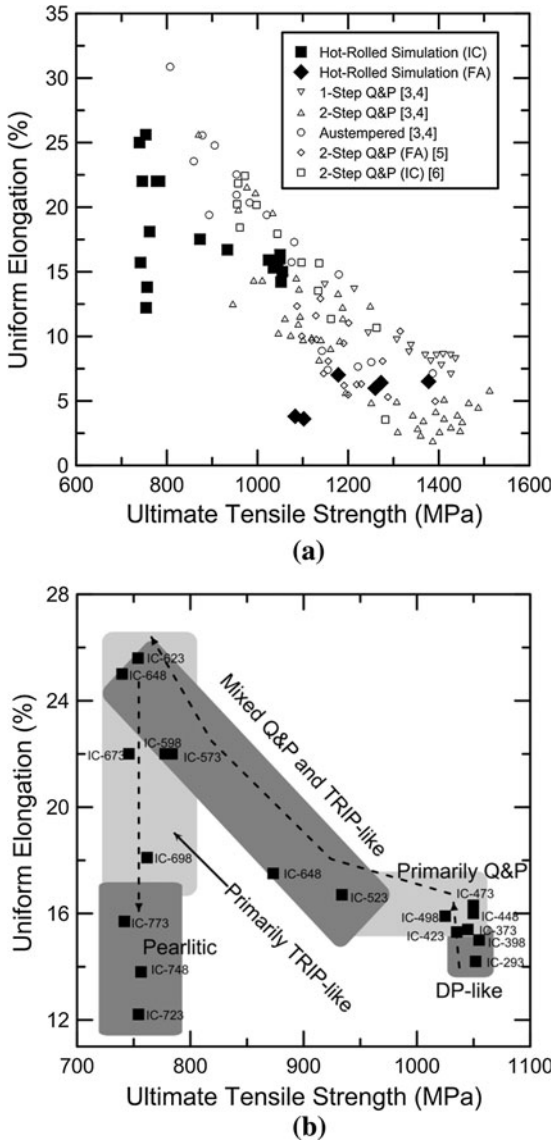


Fig. 8—(*a*) Mechanical property combinations from hot-rolling simulations and from previous Q&P literature.[3–6] (*b*) Expanded view of (a) showing only results from hot-rolling simulations of IC samples. The CT (in Kelvin) for each sample is indicated in the figure, and dashed arrows indicate the general trend with increasing CT. The shaded boxes qualitatively indicate the microstructural characterizations associated with the CTs.

Q&P and austempering methods. The dashed arrows in Figure 8(b) indicate the general trend of increasing coiling temperature. It is interesting to note that for low coiling temperatures (293 K to 498 K (20 °C to 225 °C)), the mechanical properties combinations were relatively insensitive to coiling temperature variations. Further, for the higher coiling temperatures (623 K to 773 K (350 °C to 500 °C)), the uniform elongations were significantly affected by variations in coiling temperature; however, tensile strengths did not vary with changes in coiling temperature. Finally, for the intermediate coiling temperatures (498 K to 623 K (225 °C to 350 °C)), strength and elongation both varied with coiling temperature.

IV. CONCLUSIONS

Earlier calculations suggested that producing Q&P microstructures directly from the hot-strip mill may be conceptually viable.[7] The concept involves partial martensite formation before coiling with nonisothermal carbon partitioning during cooling of the wound coil. Experimental work simulative of hot-strip mill cooling practices produced results that were similar to those of previous Q&P research that used isothermal partitioning, *i.e.*, equally high retained austenite fractions with similar microstructures and mechanical property combinations. These results confirm that Q&P microstructures may be achievable *via* hot-strip mill processing. Coiling temperature control at low temperatures and variations in the cooling profile as a function of position within the wound coil will influence property uniformity, and these issues thus warrant further attention in any implementation efforts.

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