Effect of Microstructure on Strength and Toughness of Heat-Treated Low Alloy Structural Steels

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Several low alloy structural steels with different levels of Ni, Cr, and Mo and carbon contents ranging from 0.12 to 0.42 wt pct have been studied to determine the effect of transformation structures on strength and toughness. The strength and toughness were, respectively, evaluated with the yield stress (0.2 pct proof stress) in tensile tests under ambient temperature and ductile-brittle transition temperature (DBTT) in Charpy impact tests. The significant conclusions are as follows: well-defined packets are observed in martensitic and lower bainitic structures, and in this case the packet diameter is the primary microstructural parameter controlling the yield stress and DBTT. The mechanical properties are also improved to a lesser degree with decreasing width of the lath present within the packet. If the steel has an upper bainitic structure, the packet is composed of well-defined blocks, and the block size controls the yield stress and DBTT.

I. INTRODUCTION

IT has been recognized that phase transformation products such as martensite and bainite play a dominant role in determining the mechanical properties of heat-treated low alloy structural steels. Commercial, heat-treated low alloy structural steels have martensitic and bainitic structures with a lath type substructure.¹⁻⁶ The lath type structure is composed of packets of parallel laths within the prior austenite grain. The packet consists of two components: (1) a matrix of laths separated by low-angle boundaries and (2) sets of other laths that are separated from the matrix by high angle boundaries.^{1.2.6} The matrix of laths having the low-angle boundaries will be called "a block" throughout this paper.

Many investigators have shown that prior austenite grain size plays a role in controlling the strength and toughness of steels having lath martensitic and bainitic structure. For example, a Hall-Petch type relationship was observed to exist between the prior austenite grain size and the steel's strength.^{7,8} More recently, however, it was shown that the vield stress and DBTT of martensitic and bainitic steels depend strongly on the packet diameter.^{4,9-15} On the other hand, Owen¹⁶ has suggested that the block size is the primary microstructural unit controlling the yield stress of Fe-Ni-C alloys. Matsuda et al.¹⁷ have shown that DBTT of low carbon tempered martensitic and bainitic steels depends on the covariant packet size where a covariant packet is synonymous to the block previously mentioned. Nevertheless, Smith and Hehemann¹⁸ have found that the yield stress varies inversely with the lath width in AISI 4340 steels having martensitic and lower bainitic structure. Most recently, Naylor¹⁹ has indicated that the lath width is the basic parameter controlling the yield stress and DBTT of Fe-Mn and Fe-Mn-Cr low carbon steels having lath martensitic or bainitic structures. In contrast with the previous investigations, Brozzo et al.⁵ have demonstrated that the yield stress of 2 pct Mn-3 pct Cr low-carbon bainitic steels does not change significantly with variation in prior austenite grain size or covariant packet size.

The above survey of literature serves to show the complex effects of microstructure on the mechanical properties. It is therefore the purpose of the present paper to study the effect of transformation structures on the strength and toughness of several low alloy structural steels and to find the dominant microstructural factor that controls the steel's properties.

II. EXPERIMENTAL

The steels used in this investigation were low alloy structural steels with different levels of Ni, Cr, and Mo and carbon contents ranging from 0.12 to 0.42 wt pct. The chemical compositions of the steels are given in Table I. The steels were received as 25 mm-diameter hot rolled and fully annealed bars. The heat treatments given to test steels are given in Table II. All specimens were austenitized in an argon atmosphere tube furnace with a flat zone temperature accuracy of ± 0.5 K. Quenching was done in iced brine. Lower and upper bainitic structures were produced by quenching the steels directly into a lead-tin bath followed by isothermal transformation at the required temperature above Ms. The lead-tin bath used in this investigation had a sufficiently large thermal capacity. The martensitic steels were double-tempered for 7.2 ks per temper at 473 K in an oil bath with intermediate quenching in an iced brine and refrigeration in liquid nitrogen. The purpose of the refrigeration was completely to eliminate retained austenite. Tensile specimens with a gage length of 30 mm and a cross section 7 mm-diameter were used. The tensile test was performed with an Instron machine at room temperature (293 K) at a constant strain rate of 3.35 \times 10 s⁻¹. The standard full size Charpy V-notch specimens were broken in a Charpy impact machine with a hammer velocity of 3.5 m/s. The microstructure was examined by optical metallography and thinfoil transmission electron microscopy (TEM). Specimens for optical metallography were cut from the ends of tensile and Charpy impact specimens. The specimens were first etched with a supersaturated aqueous solution of picric acid containing 0.2 pct of a surface agent, which is known to reveal the prior austenite grain size.^{21,22} Then, the specimens were etched with a supersaturated aqueous solution of picric acid containing 0.08 pct cupric chloride and 2 pct nital to delineate the lath structure within the prior austenite grains.

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Table I. Chemical Composition (Wt Pct) and Ms Point (K) of Steels Used

Designation								Ms* Austenitizing Temp.		
of Steel	C	Si	Mn	Р	S	Ni	Cr	Мо	(1133 K)	(1473 K)
Fe-0.12C-Ni-Cr	0.12	0.28	0.68	0.015	0.012	2.30	0.45		710	713
Fe-0.15C-Cr-Mo	0.15	0.27	0.70	0.017	0.014	—	1.05	0.16	695	697
Fe-0.20C-Ni-Cr-Mo	0.20	0.30	0.62	0.012	0.010	1.60	0.49	0.15	670	671
Fe-0.32C-Ni-Cr	0.32	0.25	0.70	0.011	0.010	3.35	0.88		598	602
Fe-0.37C-Cr-Mo	0.37	0.27	0.72	0.016	0.020	_	1.04	0.16	590	593
Fe-0.42C-Ni-Cr-Mo	0.42	0.27	0.73	0.010	0.006	1.73	0.81	0.16	583	585
*The Ms point was determined by standard dilatometry.										

Table II. Heat Treatment Schedule Studied

_	Designation	Heat Treatment
I.	Heat treatment for producing fully martensitic steels having the various prior austenite grain size [IBQ \rightarrow sub (77 K) plus T (473 K)]	Austenitize at 1133 to 1473 K (3.6 to 10.8 ks), quench in an iced brine, subzero-treat at 77 K (72 ks), double-temper for 7.2 ks per temper at 473 K with intermediate quenching in an iced brine and refrigeration at 77 K (72 ks).
II.	Heat treatment for producing fully lower bainitic steels hav- ing the various prior austenite grain size [IT (588 K) \rightarrow IBQ]	Austenitize at 1133 to 1473 K (3.6 to 10.8 ks), isothermally transform at 593 K (36 ks), quench in an iced brine.
III.	Heat treatment for producing fully upper bainitic steels hav- ing the various prior austenite grain size (a) [IT (673 K) \rightarrow IBQ] (b) [IT (693 K) \rightarrow IBQ]	Austenitize at 1133 to 1473 K (3.6 to 10.8 ks), isothermally transform at 673 K (108 ks) or 693 K (72 ks), quench in iced brine.

Thin foils were prepared by grinding to 0.1 mm thickness, then chemically thinning in a mixed solution by hydrofluoric acid and hydrogen peroxide, and finally, electropolishing in a mixed solution of phosphoric and chromic acids. The prior austenite grain size, packet diameter, and block size in martensitic and bainitic steels were determined by ASTM linear intercept measurements on optical micrographs. The lath widths were estimated from transmission electron micrographs. About 300 laths in each specimen were taken to measure the average lath width from the micrographs. Fractographic analysis was made on the fresh surfaces of broken impact specimens using a scanning electron microscope (SEM).

III. RESULTS AND DISCUSSION

A. Metallographic Observations

Figure 1 shows representative optical and transmission electron micrographs of martensitic steels, which were Fe-0.20C-Ni-Cr-Mo and 0.42C-Ni-Cr-Mo steels. In these steels packets were distinctly present within the prior austenite grain, but no clear regions of blocks were delineated by the present etching procedure (Figures 1(a) and (c)). TEM revealed that the martensite possessed parallel lath morphology and that the packet was composed of parallel laths (Figures 1(b) and (d)). A small amount of twin plates was observed in the steels with carbon contents greater than 0.37 pct.

Figure 2 shows representative micrographs for bainitic steels, the Fe-0.20C-Ni-Cr-Mo and Fe-0.42C-Ni-Cr-Mo steels. The lath structure of the Fe-0.42C-Ni-Cr-Mo lower bainitic steel was similar to that of martensitic steel with the same alloying elements in that the packet area is clearly

observed, but the block region is obscure (Figure 2(a)). For the Fe-0.20C-Ni-Cr-Mo and Fe-0.42C-Ni-Cr-Mo upper bainitic steels, the packet within the prior austenite grain was partitioned by blocks (Figures 2(c) and (e)). TEM revealed that for the 0.42C-Ni-Cr-Mo lower bainitic steel, the packet was composed of parallel laths in which the internal carbide precipitates were oriented in one direction (Figure 2(b)). On the other hand, for the Fe-0.20C-Ni-Cr-Mo and Fe-0.42C-Ni-Cr-Mo upper bainitic steels, the packet consisted of parallel laths with internal carbide stringer lying along the length of the laths (Figures 2(d) and (f)).

B. Mechanical Properties

The relationships between the yield stress $(\sigma_{0.2})$ and the reciprocals of the square root of the prior austenite grain size $(d_p^{-1/2})$ and packet diameter $(d_p^{-1/2})$ for various martensitic steels are shown in Figures 3 and 4. The yield stress depended on the packet diameter rather than the prior austenite grain size. A Hall-Petch type relation, $\sigma_{0.2} = \sigma_i + k_y d_p^{-1/2}$, where σ_i and k_y are material constants, was found between the yield stress and the packet diameter. No such linearity was obtained between $\sigma_{0.2}$ and $d_y^{-1/2}$. A least square analysis provided the values for σ_i and k_y shown in Table III.

It is significant to note that the values of k_y are comparable to those for iron and steels with 0.0014 to 0.15 pct carbon as reported by Gouzou.²³ This similarity between the values of k_y for the present martensitic steels and Gouzou's results suggests that the martensite packet boundary provides a strengthening effect comparable to the grain boundary strengthening effect in ferrite.



Fig. 1 — Optical and transmission electron micrographs of lath structure in martensitic steels. (a) and (b) are for Fe-0.20C-Ni-Cr-Mo steel heat-treated by a schedule I (austenitized at 1473 K) in Table II. (c) and (d) are for Fe-0.42C-Ni-Cr-Mo steel heat-treated by a schedule I (austenitized at 1473 K) in Table II. A and P indicate prior austenite grain boundary and packet area, respectively.

Table III. Values of σ_i and k_y in the Relation, $\sigma_{0.2} = \sigma_i + k_y d_p^{-1/2}$, for Martensitic Steels

Designation of Steel	σ_i (MPa)	$\frac{k_y}{(\text{MPa}/\text{mm}^{-1/2})}$		
Fe-0.12C-Ni-Cr	830	20.8		
Fe-0.15C-Cr-Mo	834	25.7		
Fe-0.20C-Ni-Cr-Mo	838	34.1		
Fe-0.32C-Ni-Cr	1035	39.2		
Fe-0.37C-Cr-Mo	1150	38.8		
Fe-0.42C-Ni-Cr-Mo	1264	40.1		

Another marked fact is that the values of k_y increase with decreasing lath width owing to a lower Ms temperature. The values of k_y increased linearly with the reciprocal <u>of</u> the square root of the arithmetic mean of the lath width $(W_L^{-1/2})$ (Figure 5). In this regard, Porter and Dabkowski⁸ have shown that the decrease in the number of ferrite grains or lath increases the values of k_y when a 5Ni-Cr-Mo-V was treated with rapid heat treatment. Kosik *et al.*²⁴ have also demonstrated that in hot-worked Armco iron, silicon steel, and aluminum metal, the values of k_y increase with the inverse of subgrain size. Thus, the decrease in the lath width of martensitic steels adds additionally to the yield stress.

Figure 6 shows the relationship among $\sigma_{0.2}$, $d_{\gamma}^{-1/2}$, and $d_p^{-1/2}$ for various bainitic steels. For the Fe-0.42C-Ni-Cr-Mo lower bainitic steel, the yield stress depends on the packet

diameter following a Hall-Petch type relationship. For the Fe-0.20C-Ni-Cr-Mo and Fe-0.42C-Ni-Cr-Mo upper bainitic steels, however, small increases in $\sigma_{0,2}$ were observed despite a significant decrease in the prior austenite grain size and packet diameter. The changes in the 0.2 pct proof stress, owing to variations of the prior austenite grain size, are similar to the alterations of the block size accompanying the prior austenite grain size. For the Fe-0.20C-Ni-Cr-Mo upper bainitic steel, the average block size increases by only 1.6 μ m even when the prior austenite grain size increases from 85 to 198 µm. For the Fe-0.42C-Ni-Cr-Mo upper bainitic steel, the average block size increases by only 5.2 μ m even when the prior austenite grain size varies from 67 to 412 μ m. This similarity between the changes in the 0.2 pct proof stress and the alterations of the block size suggests that for the upper bainitic steels, the bainitic block boundary provides a significant strengthening effect. There is, however, literature discussing that the small increase in the yield stress of the upper bainitic steel is affected by the lath structure within the block. Brozzo et al.⁵ have shown that the small increase in the values obtained for the yield stress of 2 pct Mn-3 pct Cr low-carbon bainitic steel is attributed to the lath structure derived from very fine austenite grain. For upper bainitic steels studied in this paper, a significant difference in the lath width is not observed despite large differences in grain size. The above results along with the metallographic examination shown in



Fig. 2—Optical and transmission electron micrographs of lath structure in various bainitic steels. (a) and (b) are for Fe-0.42C-Ni-Cr-Mo steel heat-treated by a schedule II (austenitized at 1473 K) in Table II. (c) and (d) are for Fe-0.20C-Ni-Cr-Mo steel heat-treated by a schedule III-(b) (austenitized at 1473 K) in Table II. (e) and (f) are for Fe-0.42C-Ni-Cr-Mo steel heat-treated by a schedule III-(a) (austenitized at 1473 K) in Table II. (e) and (f) are for Fe-0.42C-Ni-Cr-Mo steel heat-treated by a schedule III-(a) (austenitized at 1473 K) in Table II. And PB indicate prior austenite grain and packet boundary, respectively. P and B show packet area and block region, respectively.

Figures 1 and 2 indicate that the packet boundaries have strong strengthening effect and lath boundaries have additional strengthening effect and that, if the packet is composed of the blocks partitioning the packet as in the upper bainitic steels, the block boundaries have a significant strengthening effect.

Figures 7 and 8 show the relationship among the DBTT, $d_{\gamma}^{-1/2}$, and $d_{p}^{-1/2}$ for various martensitic and bainitic steels. Sharp transition between ductile and brittle fracture was observed over a narrow temperature range in the martensitic and bainitic steels with carbon contents of no more than 0.20 pct. Martensitic and bainitic steels with carbon contents of 0.42 pct showed fracture which had an intermediate energy absorption. In the latter steels, therefore, the DBTT was defined as the temperature which shows intermediate energy absorption between the upper and lower shelf. The results obtained are (1) the DBTT of martensitic and lower bainitic steels decreased linearly with increase in $d_p^{-1/2}$ (Figures 7 and 8), but such linearity was not obtained between the DBTT and $d_{\gamma}^{-1/2}$; (2) for martensitic steels, the slope increased with decreasing lath width; and (3) for upper bainitic steels, a slightly decreased DBTT was observed despite a significant increase in $d_{\gamma}^{-1/2}$ and $d_p^{-1/2}$ (Figure 8).

C. Fractography

Figures 9 and 10 show typical fractured surfaces of impact specimens broken at temperatures below the DBTTs of



Fig. 3—Effect of prior austenite grain size and packet diameter on yield stress of martensitic steels with carbon contents ranging from 0.12 to 0.20 pct.



Fig. 4—Effect of prior austenite grain size and packet diameter on yield stress of martensitic steels with carbon contents ranging from 0.32 to 0.42 pct.

various martensitic and bainitic steels. As expected, a similar fracture appearance was found between the martensitic and lower bainitic steels. Fractured surfaces of the martensitic and lower bainitic steels consisted of quasicleavage facets of the order of the packet diameter. Complex river patterns, consisting of small cleavage steps, were observed within the facets (Figure 9). Here, a quasicleavage facet is



Fig. 5—Effect of the lath width on k_y parameter in the Hall-Petch equation.



Fig. 6— Effect of the prior austenite grain size and packet diameter on the yield stress of various bainitic steels.



Fig. 7—Effect of the prior austenite grain size and packet diameter on the DBTT of various martensitic steels.



Fig. 9—Fracture surfaces of martensitic and lower bainitic steels at temperatures below DBTT. (a) Fe-0.20C-Ni-Cr-Mo steel heat-treated by a schedule I (austenitized at 1473 K) in Table II. (b) Fe-0.42C-Ni-Cr-Mo steel heat-treated by a schedule I (austenitized at 1473 K) in Table II. (c) Fe-0.42C-Ni-Cr-Mo steel heat-treated by a schedule II (austenitized at 1473 K) in Table II. QF indicates a quasicleavage facet.



Fig. 8-Effect of the prior austenite grain size and packet diameter on the DBTT of various bainitic steels.



Fig. 10—Fractured surfaces of upper bainitic steels at temperatures below the DBTT. (a) Fe-0.20C-Ni-Cr-Mo steel heat-treated by a schedule III-(b) (austenitized at 1473 K) in Table II. (b) Fe-0.42C-Ni-Cr-Mo steel heattreated by a schedule III-(a) (austenitized at 1473 K) in Table II. CF indicates a cleavage facet.

Steel and Structure	Austenitizing Condition K (ks)	Packet Diameter (µm)	Block Size (µm)	Arithmetic Mean of the Quasicleavage or Cleavage Facet Size, (μm)
Fe-0.20C-Ni-Cr-Mo	1323 (7.2)	96.8		110.8
(martensite)	1473 (3.6)	54.0		60.5
Fe-0.42C-Ni-Cr-Mo	1323 (7.2)	35.8	_	38.5
(martensite)	1473 (3.6)	78.9		89.3
Fe-0.42C-Ni-Cr-Mo	1323 (7.2)	32.8		39.6
(lower bainite)	1473 (3.6)	81.0		100.8
Fe-0.20C-Ni-Cr-Mo	1323 (7.2)	32.1	1.6	2.0
(upper bainite)	1473 (3.6)	88.0	3.1	3.8
Fe-0.42C-Ni-Cr-Mo	1323 (7.2)	35.0	5.2	7.1
(upper bainite)	1473 (3.6)	78.5	10.3	12.9

Table IV. Relationship between the Packet Diameter and Block Size and Arithmetic Mean of the Quasicleavage or Cleavage Facet Size for Martensitic and Bainitic Steels

defined as the spacing divided by heavy tear lines as shown by arrows in Figure 9. On the other hand, a fracture mode different from those for martensitic and lower bainitic steels was observed for the upper bainitic steels. Fractures of the upper bainitic steels were composed of the small cleavage facets with a spacing of the order of the block size, as shown by arrows in Figure 10. The arithmetic mean of the observed facet size was determined by ASTM linear intercept method. The results are given in Table IV. From these results, the arithmetic mean of the quasicleavage facet size of the martensitic and lower bainitic steels was found to correspond to the packet diameter. On the other hand, the arithmetic mean of the cleavage facet size of the upper bainitic steels was found to be almost equivalent to their block size. This close agreement strongly supports the fact that the primary microstructural parameter controlling the DBTT of the martensitic and lower bainitic steels is the packet diameter. The basic parameter controlling the DBTT of the upper bainitic steels is the block size.

IV. CONCLUSIONS

In martensitic and lower bainitic steels "packets" are the primary constituent within the prior austenite grains, and the strength and toughness of the steels are improved with decrease in the packet diameter. In upper bainitic steels "blocks" rather than packets are the dominant feature of the microstructure, and the blocks have an effect on the strength and toughness properties similar to the packets in martensitic and lower bainitic steels.

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