Influence of Cooling Rate on the Microstructure and Retained Austenite in an Intercritically Annealed Vanadium Containing HSLA Steel

NARASIMHA-RAO V. BANGARU and ANIL K. SACHDEV

The influence of cooling rate from the intercritical $\alpha + \gamma$ region on the microstructure of a vanadium bearing HSLA steel was investigated by transmission electron microscopy. Oil quenching produced an essentially ferrite-martensite dual phase structure with ~4 vol pct of fine particle and thin film retained austenite. In contrast, the slower air cooling resulted in a larger amount (~10 vol pct) of retained austenite in addition to the ferrite and martensite phases. A major portion of the retained austenite in the air cooled specimen was of the blocky morphology (1 to 6 μm), with the remainder being the submicron variety, similar to that of the oil quenched specimen. In conformance with the terminology of earlier studies, "retained" ferrite and "transformed" ferrite were observed in the air cooled steel while oil quenching completely suppressed the transformed ferrite. Retained ferrite, the cleaner of the two in terms of precipitate content, is the high temperature ferrite that coexists with the austenite at the intercritical temperature and which is retained on cooling. The transformed ferrite, on the other hand, forms from the decomposition of the austenite and contains banded carbonitrides (row precipitation) much like the initial microstructure of the HSLA steel.

I. INTRODUCTION

THE recently developed "dual phase" steels[1-5] exhibit far superior strength/ductility combinations compared to the high strength, low alloy (HSLA) steels of identical composition. Dual phase steels are "natural composites" produced by the exploitation of solid-state phase transformations and typically consist of a strong martensite phase finely dispersed in a soft ferrite matrix. However, many steels described as "dual phase" actually deviate substantially from a strictly ferrite-martensite microstructure and have been reported to contain as much as 8 vol pct of fine (submicron), blocky retained austenite.[6,7] Although the emphasis has centered initially on the ferrite and martensite, the importance of retained austenite on the mechanical properties as well as its characterization are the focus of some recent studies.[6,7,8]

One of the important processing variables for the production of dual phase steels is the cooling rate following intercritical annealing. (See, for example, the conference proceedings listed in References 3 and 9.) Rashid[3] showed the strength-ductility combination to be optimum around 8 °C per second (for 2.5 mm section) and decrease with either faster or slower cooling rates. The purpose of the present study was to characterize in detail, by transmission electron microscopy, the microstructure of an intercritically annealed vanadium containing dual phase steel (see Table I for composition) but which had been cooled at different rates. Two samples were obtained: one was air cooled (6 °C per second) and the other oil quenched (14 °C per second) following a four minute intercritical anneal at 1450 °F (788 °C). Stress strain curves of samples of these two steels are shown in Figure 1.

II. EXPERIMENTAL PROCEDURE

Transmission electron microscopy specimens were obtained from heat treated tensile blanks. About 2.5 cm long pieces were cut from the gage sections of the 2 mm thick × 12.8 mm wide tensile blanks. These sections were surface ground to 0.625 mm with flood cooling, and material was removed equally from both sides, about 0.012 mm at a time. Chemical thinning in a solution containing 5 pct HF in H_2O_2 (30 pct concentration) was used to reduce the thickness to 0.125 mm. Final electropolishing was done in a Fischione twin jet apparatus at room temperature using a chromic-acetic acid solution. Thin foils so obtained were examined in a JEOL JEM 200C transmission/scanning transmission electron microscope equipped with a Kevex energy dispersive X-ray analyzer at an accelerating voltage of 200 KV. Microchemical analysis was performed in the STEM mode of operation by mounting the specimens in a graphite holder and using a counting time of 60 seconds at 200 KV. It was established from the contamination spots that there was negligible change in foil thickness between the many ferrite and austenite phase regions used for manganese partitioning studies. The thickness of the foil in regions used for STEM analyses was around 100 nm. The beam size for the STEM probe on the specimen surface was approximately 5 nm with an effective analysis volume of 20 nm in diameter and the thickness of the foil. The error bars in the concentration plots indicate the precision (one standard deviation) as determined from the total counts on

NARASIMHA-RAO V. BANGARU (formerly known as B. V. N. Rao), formerly with General Motors Research Laboratories, Warren, MI, is now Staff Engineer, Exxon Research and Engineering Company, P.O. Box 101, Building SP-240, Florham Park, NJ 07932. ANIL K. SACHDEV is Staff Research Engineer in the Metallurgy Department, General Motors Research Laboratories, Warren, MI 48090.

Table I. Chemical Composition (Wt Pct) of Steel Investigated[3]

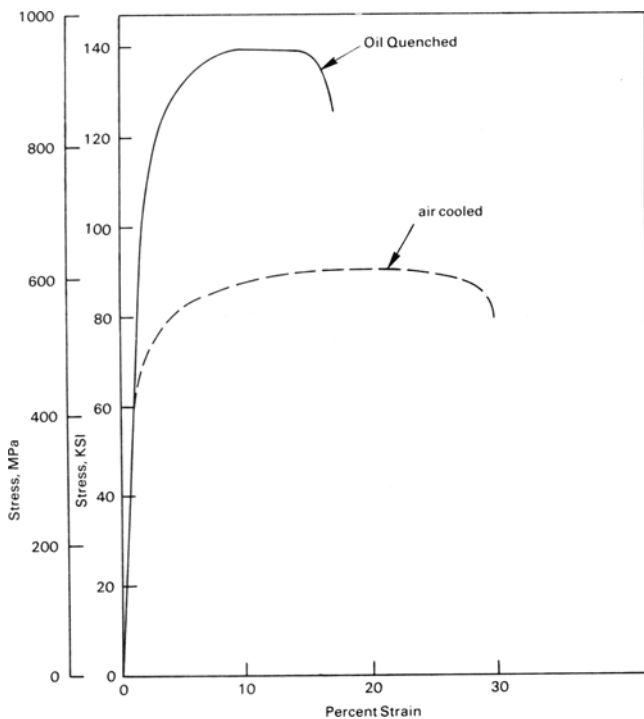| C	Mn	Si	V	N	Al	P	S
0.12	1.44	0.52	0.13	0.0196	0.077	0.008	0.011

Fig. 1 — Stress-strain curves for air cooled and oil quenched "dual-phase" steels.[1] Strain indicated is for 50.8 mm (2 in.) gage length specimens.

the elemental peaks and the adjacent backgrounds. The compositions were determined by the method of Schreiber and Wims,[10] and the influence of the Mn K_β line on the Fe K_α line was ignored because it is negligible at Mn concentrations below 5 pct.

Volume fractions of various phases were roughly estimated from a sufficient number of bright-field and dark-field transmission electron micrographs using the linear intercept method, and the amount of austenite later confirmed by X-ray analysis.

III. RESULTS

A. Ferrite

Oil Quench. This specimen contained approximately 50 vol pct retained ferrite* (Figure 2(a)). It was frequently

---

*Retained ferrite, first proposed by Krauss,[11] is the ferrite that coexists with austenite at the end of intercritical annealing and is retained on cooling to room temperature.

---

characterized by a high dislocation density particularly close to the ferrite/martensite interface. Numerous slip traces indicated that this ferrite was extensively deformed and that several of the accommodation dislocations introduced in this phase during processing were highly mobile. The weak beam dark field (WBDF) micrograph, Figure 2(b), revealed no evidence of fine precipitation at these dislocations. However, a few coarse undissolved vanadium carbonitride precipitates, V(CN), about 4 to 20 nm in diameter, were observed in this ferrite.

Air Cool. This specimen contained a much higher total volume fraction of ferrite, about 75 vol pct, compared to the oil quenched specimens. However, the ferrite in this specimen was made up of two distinctly different types: retained and "transformed" ferrites. The "transformed" ferrite was
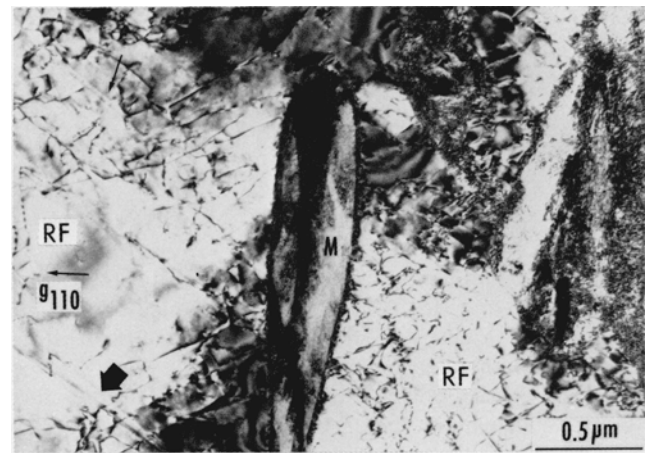
(a)
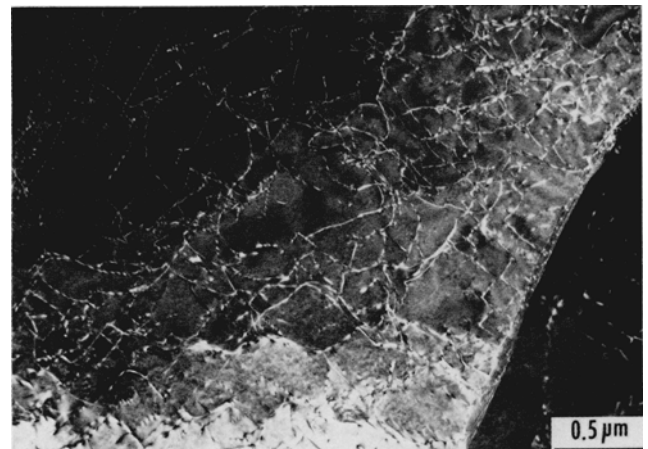
(b)

Fig. 2 — (a) Bright field micrograph revealing the high density of accommodation dislocations close to the retained ferrite (RF)/martensite (M) interface in the oil quenched specimen. Arrow shows the slip traces in ferrite. (b) Weak beam dark field ($\vec{g}$, 3$\vec{g}$) micrograph from a different area of retained ferrite showing no precipitation at the accommodation dislocations of the oil-quenched specimen.

the product of the continuous eutectoid transformation of some of the austenite during the slow cool from the two-phase α-γ field and hence the name "transformed" ferrite.

The vol pct of retained ferrite was identical in the air cooled and oil quenched specimens, but there were differences in the substructure. One difference noted was the lower density of accommodation dislocations in the retained ferrite of the air cooled specimen (Figure 3). Additionally, the dislocations were much less tangled (compare Figures 2 and 3). Undissolved alloy carbonitrides similar to those in the oil quenched specimen were also found.

Transformed ferrite in the air cooled specimen comprised about 25 vol pct of the microstructure. This ferrite was not observed in the oil quenched specimen and resulted from the decomposition of austenite by an interphase precipitation mechanism.[12] The transformed ferrite was seen to contain V(CN) precipitates in rows (Figure 4). The rows had an average spacing of 30 nm and, in a row, the particles were separated by about 20 nm. The carbonitrides in the well defined rows (arrows 3, Figure 4(a)) were the finest (average diameter 7 nm) and were coherent as indicated by
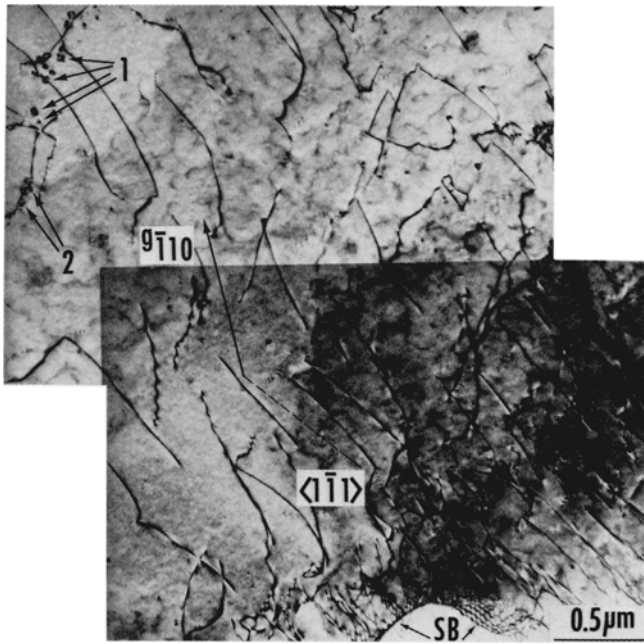
Fig. 3—Bright field micrograph showing accommodation dislocations in the retained ferrite of air cooled specimen. Arrows 1 show partially coherent/incoherent vanadium carbonitrides while arrows 2 reveal the highly coherent carbonitrides.

the double arc strain contrast. Precipitates identified by arrows 2 were also aligned carbonitrides which formed on cooling but were larger (average diameter 17 nm) because these nucleated heterogeneously at the low angle sub-boundaries of the original austenite and hence grew more rapidly due to the enhanced diffusion at the subboundaries. This process apparently led to solute depletion in the immediate vicinity of the subboundary as evidenced by a lack of precipitation in this region (Figure 4(a)). The coarsest and isolated carbides (average diameter 40 nm, arrow 1) were identified as carbonitrides that were undissolved in the austenite and grew at the intercritical temperature. These were then retained in the ferrite on cooling. Because of a change in diffraction conditions due to a small misorientation across the subboundary, carbonitrides in region N (Figure 4 (a)) are out of contrast.

B. Martensite

Oil Quench. About 50 vol pct of the microstructure in this specimen consisted of the highly dislocated lath martensite (Figure 5). Some auto-tempered cementite was also observed within the martensite laths (not shown) indicating a high M_s temperature of about 250 °C for this martensite. In addition, about 1 vol pct of interlath thin film retained austenite was also identified as seen in the DF micrograph, Figure 5(b). The morphology and substructure of this lath martensite suggested a carbon content of about 0.35 wt pct.[13]

Air Cool. The second phase in this specimen was comprised of a mixture of bainite (Figure 9) and twinned plate martensite (Figure 6). The total volume fraction of this mixture was estimated to be about 15 vol pct. This lower volume fraction martensite also produced a lower density of accommodation dislocations in the retained ferrite. The twinned martensite was in contrast to the dislocated lath
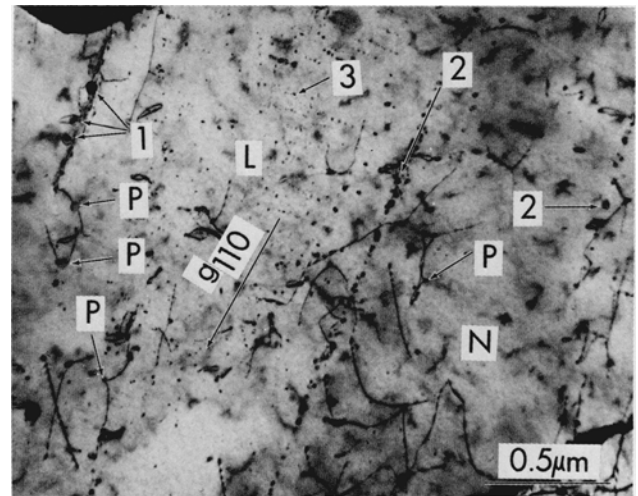

(a)
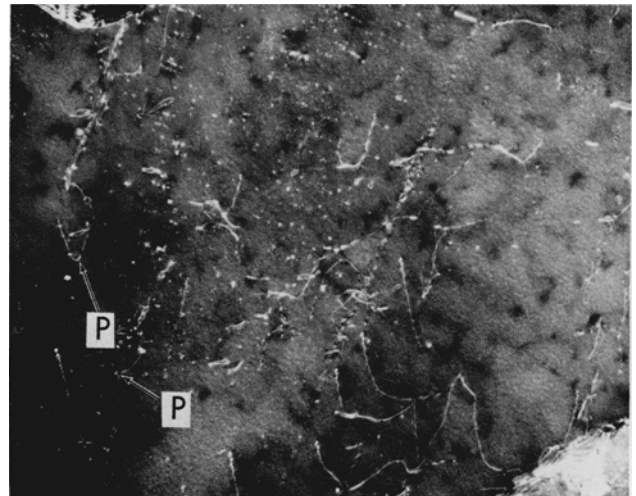
(b)

Fig. 4 — (a) Bright field micrograph revealing the transformed ferrite in air cooled specimen. Carbonitride precipitation in rows is shown by arrow 3. Arrows 1 show coarse undissolved alloy carbonitrides, and arrows 2 point to the medium sized carbonitrides precipitated at low angle boundaries, for example, separating regions L and N. Arrows P reveal dislocations pinned by precipitates. (b) The corresponding ($\bar{g}$, 3$\bar{g}$) WBDF micrograph.

martensite for the similarly heat treated but oil quenched specimen. Auto-tempered cementite was not observed in this martensite, which is consistent with the fact that twinned martensite has a lower M_s temperature than dislocated lath martensite. The lower temperature twinned martensite suggests that the austenite in this specimen was enriched in solute (>0.5 wt pct C) prior to the martensite transformation.[13]
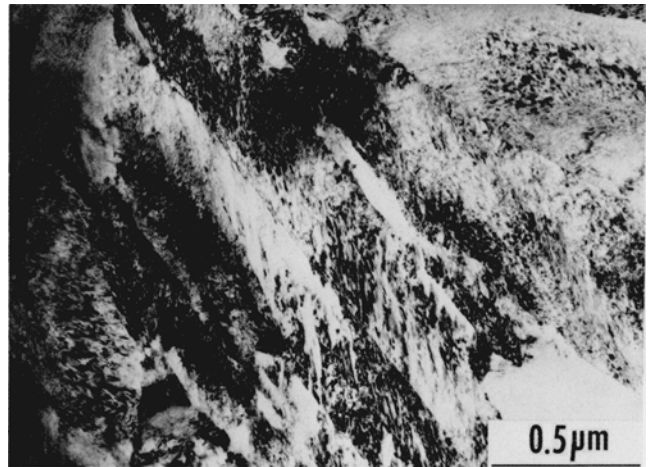
C. Retained Austenite

The retained austenite described in this section has a blocky morphology and is not the thin film austenite described in connection with the lath martensite of the oil quenched specimen.
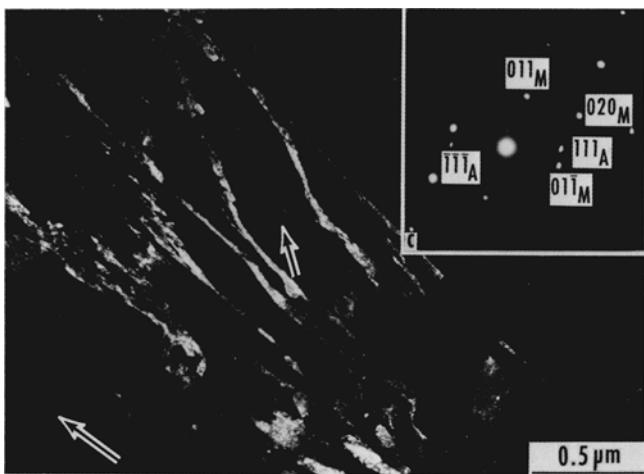
Oil Quench. About 3 vol pct fine particle austenite was estimated in this specimen in addition to the thin film austenite and was seen to be distributed at and within the ferrite
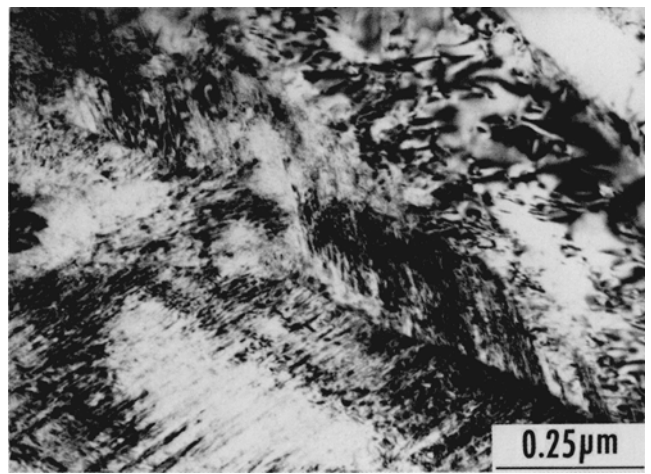
(a)

(a)
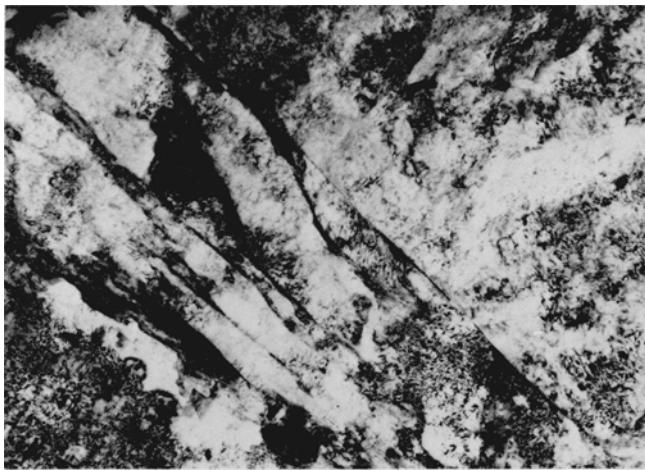
(b)

Fig. 5 — (a) Bright field, (b) dark field, and (c) selected area diffraction pattern (inset) revealing the interlath films of retained austenite in the oil quenched specimen.


(b)

Fig. 6 — Bright field micrographs of martensite from different areas of air cooled specimen showing {112} substructural twinning and adjacent twin related plates.

grain boundaries (Figure 7(a)). Figure 7(b) shows the Mn and Si concentration profiles in this austenite. These austenite particles are about 0.16 μm in diameter and are suspected to have formed from the isolated, coarse carbides of the parent microstructure during the intercritical annealing. Rigsbee[14] discussed the stabilization mechanisms for this austenite. He attributed part of the stabilization to the coherent fcc:bcc interface which he indicated provided a barrier for the shear transformation and indirectly served to increase the degree of chemical partitioning by preventing the growth of austenite. The absence of any substructure in these retained austenite particles suggests that they are very stable. These finely distributed submicron austenite particles are expected to have a high solute content because of their limited growth. Their M_s temperature is expected to be much below room temperature leading to extreme stability with respect to transformation with strain.[7,8]

Air Cool. In addition to the 3 vol pct submicron retained austenite as observed in the oil quenched specimen, about 7 vol pct of micron sized blocky retained austenite was estimated in the air cooled specimen. The additional retained austenite varied in diameter from 1 to 6 μm and

shared interfaces with the retained ferrite, transformed ferrite, or martensite (bainite). Examples of these are shown in Figures 8 through 10. The accommodation substructure in the austenite is seen to consist essentially of stacking faults. The large amount of retained austenite in the air cooled specimen was perhaps the most significant difference between the two cooling rates studied.

Manganese concentration was determined by STEM microchemical analysis across several austenite/ferrite interfaces of the blocky retained austenite and was found to be significantly heterogeneous within a particle as well as from one particle to another. Concentrations in the austenite generally varied between 1.7 and 2.3 pct Mn compared to the 1.44 Mn of the base alloy (Figures 11(a),(b)), and occasionally abnormally high concentrations of manganese were noted (Figures 11(c),(d)). It is unclear what causes these high manganese concentrations, but it is speculated that the macroscopic inhomogeneities in the original ingot (banding) could be responsible for the observed variations. The fine, submicron size particles of austenite observed in both the oil quenched and air cooled specimens also consistently revealed a substantial manganese enrichment (~2 wt pct)
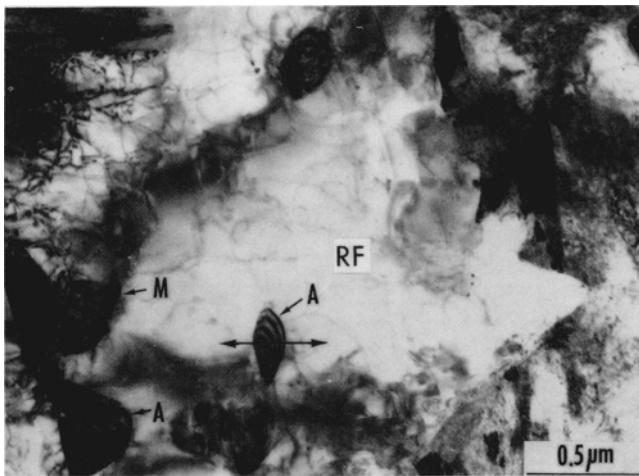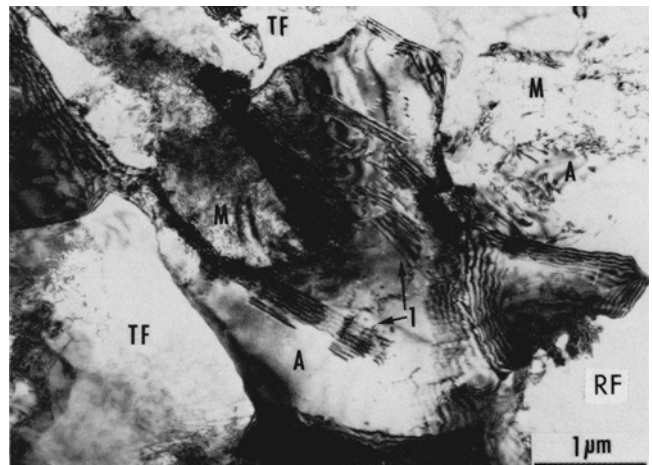
(a)
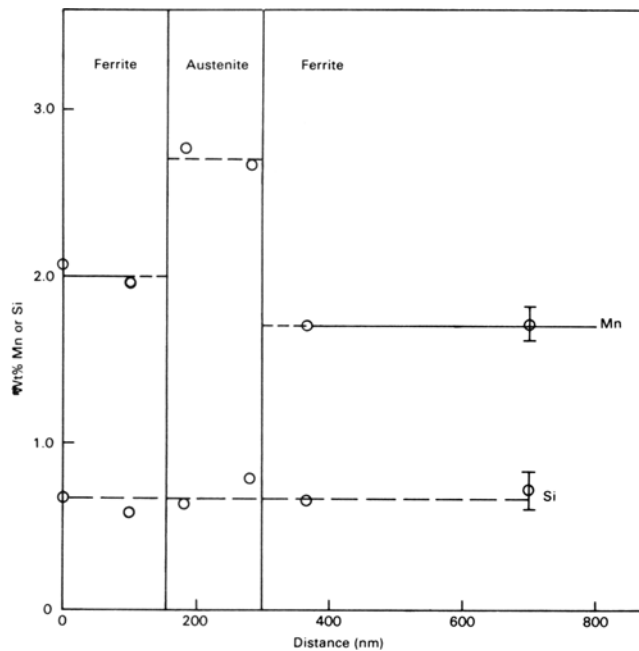
(b)

Fig. 7—Microanalysis of Mn and Si partitioning in the fine particle retained austenite of the oil quenched specimen. (a) Bright field micrograph revealing the fine particle austenite and the region used for obtaining the concentration profile shown in the plot of (b). 'M' shows the martensite, 'A' the retained austenite, and 'RF' the retained ferrite.

(Figure 7 (b)). Due to the short annealing times (three to four minutes) in the intercritical region and the relatively slower diffusion of manganese, the observed manganese partitioning must be largely the result of inheritance from the initial partitioning in the parent microstructure.

IV. DISCUSSION

A. Effect of Cooling Rate on Microstructure

The approximate amounts of the microconstituents present at room temperature following air cooling and oil quenching are summarized below:

Air Cool: Retained ferrite (50 vol pct)+Transformed ferrite (25 vol pct)+Martensite (Bainite) 15 vol pct + Retained austenite (10 vol pct)
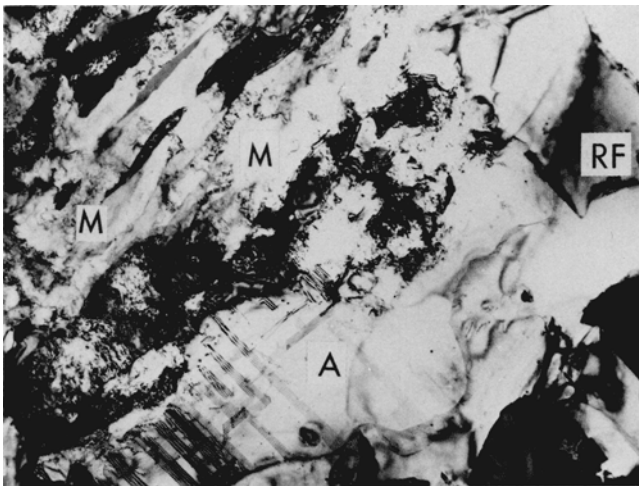

(a)
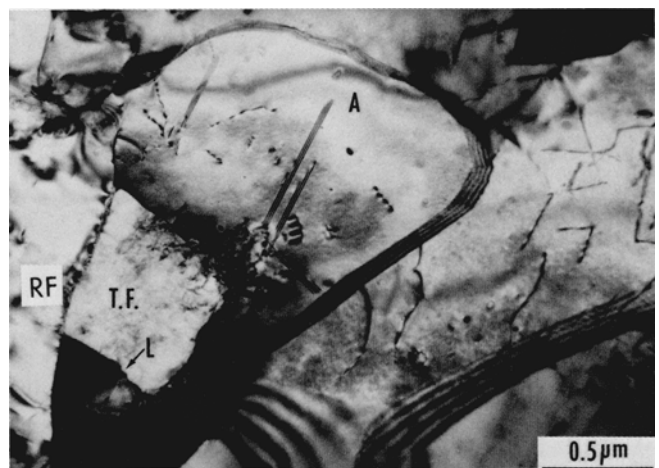
(b)

Fig. 8—(a) Bright field micrograph showing the martensite (bainite) (M)/austenite (A) transformation front in air-cooled specimen. Arrows 1 show accommodation stacking faults in the austenite. (b) Bright field micrograph from air cooled specimen showing retained austenite partially decomposed to transformed ferrite (T. F.). 'L' shows the interfacial microledges.

Oil Quench: Retained ferrite (50 vol pct) + Martensite (50 vol pct) (Retained austenite ~3 vol pct)

The high temperature microstructure in the $(\alpha + \gamma)$ region before cooling is identical for both cooling rates, namely, about 50 vol pct ferrite and 50 vol pct austenite. The 50 vol pct high temperature ferrite is retained at room temperature with either cooling rate and hence the term "retained" ferrite. The 50 pct austenite transforms completely to martensite as in the oil quenched case or to ferrite ("transformed") and twinned martensite as in the air cooled case, with some austenite being retained in the latter cooling rate.
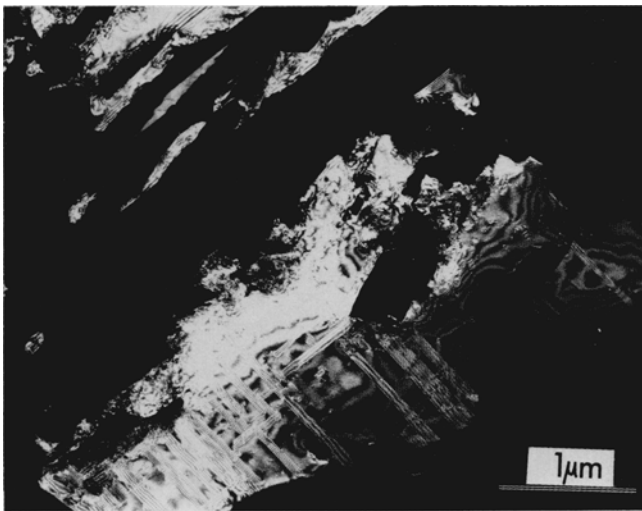
Cooling rate has important effects on (1) the isothermal (nonmartensitic) decomposition products, and (2) on interstitial solute partitioning. Oil quenching essentially suppresses both these reactions, and the entire 50 vol pct austenite undergoes diffusionless transformation to martensite. With the slower air cooling, however, decomposition
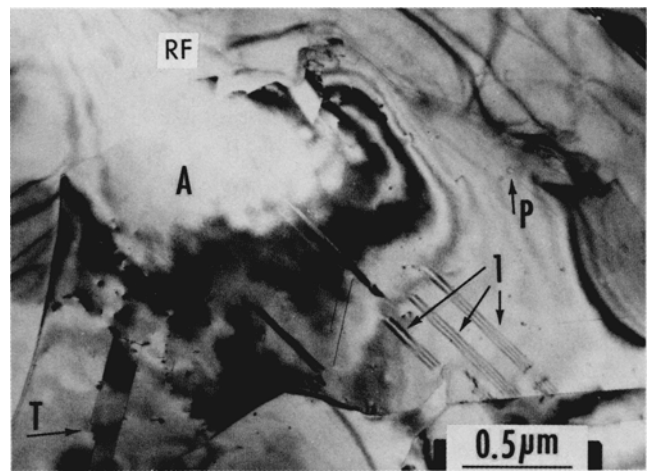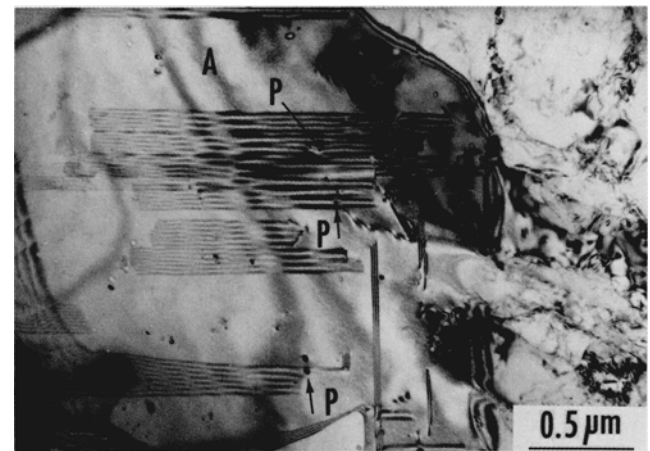
(a)

(b)

Fig. 9—Retained austenite in air cooled specimen characterized by martensite (bainite) (M)/austenite (A) transformation front. (a) Bright field and (b) the corresponding austenite dark field micrographs revealing the large volume fraction of austenite present. Both micrographs show more than 1 variant of the accommodation stacking faults.


(a)

(b)

Fig. 10—Bright field micrographs from different areas revealing the blocky retained austentie (A) in air cooled specimen. In (a), arrows 1 reveal stacking faults and T, annealing twins. In (a) and (b), 'P' indicates undissolved alloy carbonitrides pinning stacking faults.

products at high temperature cannot be suppressed and part of the austenite transforms to ferrite, strengthened by a banded carbonitride precipitation.

During air cooling, the austenite remaining after the formation of transformed ferrite should transform to martensite (and bainite). However, about 10 vol pct of blocky (1 to 6 μm) retained austenite is observed at room temperature (Figures 8 through 11). This can be explained by the fact that during the relatively slower air cooling, substantial migration of carbon (and nitrogen) into the austenite probably occurs across the austenite/ferrite interfaces. This carbon partitioning increases the hardenability of the austenite, thereby progressively restricting transformed ferrite formation and also lowering the M_s temperature, causing the relatively richer areas to be retained at room temperature. Because of the short annealing times and possible manganese banding in the steel,[15] the austenite composition is heterogeneous, and the carbon partitioning during the slow

cooling further accentuates this heterogeneity. All these serve to provide austenitic areas of different stabilities. The carbon partitioning can be indirectly supported by the fact that no twinned martensite (higher carbon) was seen in the oil quenched specimen.

B. Structure-Property Correlations

The preceding results provide insight into: (1) origin of superior uniform elongation-tensile strength combinations in the air cooled steel and (2) the origin of the low yield strength in the air cooled steel (Figure 1). The unique aspects of the air cooled microstructure are the large amounts of retained austenite and transformed ferrite. The presence of retained austenite could be very beneficial since it can transform to martensite on straining[16] and contribute to uniform elongation through the transformation induced plasticity (TRIP) phenomenon.[6] In this context, it is very significant that due to its heterogeneous chemistry and size distribution, the individual particles of retained austenite in the air cooled steel will have different stabilities and therefore undergo the martensitic transformation to provide
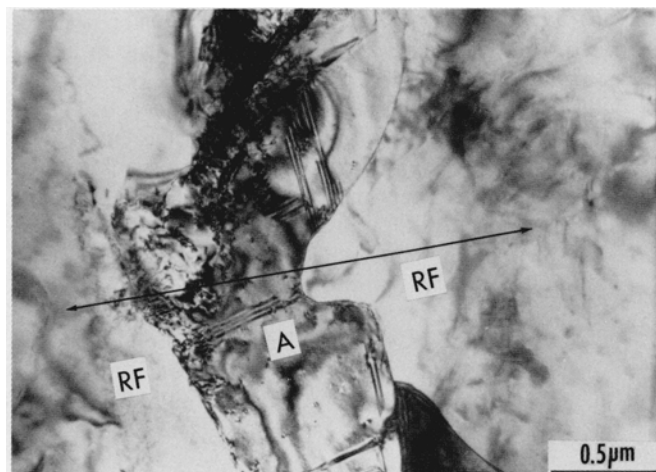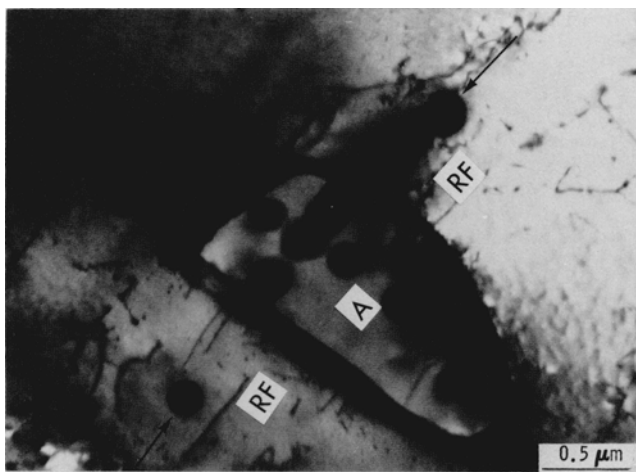
(c)
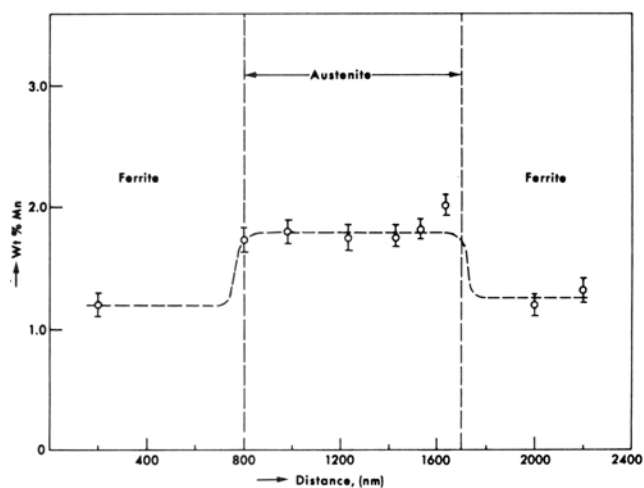
(b)
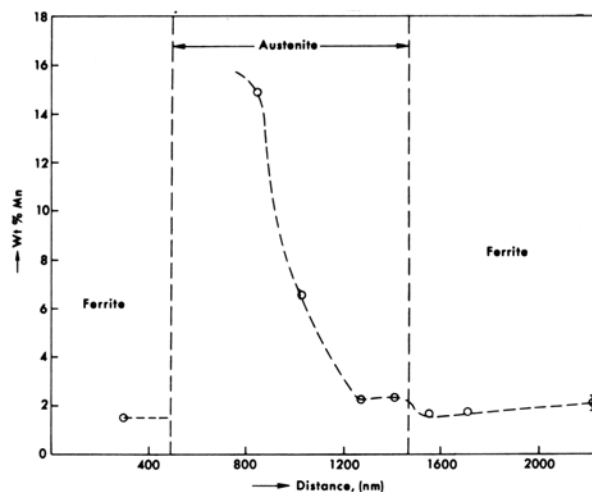
(d)

Fig. 11—Solute partitioning across retained austenite in air cooled specimen. (a) Bright field micrograph showing the traverse used for obtaining the concentration profile plotted in (b) (error bars indicate precision). (c) is the bright field micrograph corresponding to the concentration profile plotted in (d).

plasticity over a range of initial strains rather than at one time.[7,8] The low yield strength and high initial strain hardening obtainable in this steel can be explained in part by the retained austenite transformation to martensite and the resultant strengthening of the adjacent ferrite.[7] The stress induced transformation of the retained austenite could trigger flow in the surrounding ferrite at a lower stress than would otherwise be possible. This is also supported by the observation that a strictly ferrite-15 wt pct martensite steel has a higher yield strength than that of the air cooled steel (also with 15 vol pct martensite).[5]

The transformed ferrite could be beneficial because it has a higher strength than the retained ferrite and could provide an intermediate load transfer step before deformation is induced in the martensite. The strength of this ferrite is attributed to the resistance to dislocation motion provided by the aligned banded carbonitrides.

The presence of a lower amount of martensite with air cooling provides an advantage, since martensite formation is associated with a substantial volume change, and a large 50 vol pct martensite as in the oil quenched specimen requires a greater accommodation in the ferrite than

the 15 vol pct as in the air cooled specimen. The larger accommodation renders the ferrite significantly deformed and preyielded as in the oil quenched case, and reduces its formability.

V. CONCLUSIONS

1. Air cooling the intercritically annealed vanadium containing HSLA steel used in this study resulted in a triplex microstructure consisting of about 50 vol pct retained ferrite, 25 vol pct transformed ferrite, 15 vol pct martensite (bainite), and 7 vol pct blocky retained austenite. The faster oil quench prevented the formation of the transformed ferrite, and the microstructure consisted of only 50 vol pct retained ferrite and 50 vol pct martensite. About 3 vol pct of fine particle retained austenite was observed with both cooling rates.
2. The large amount of blocky (1 to 6 μm) retained austenite in the air cooled steel was concluded to be primarily the result of enhanced chemical stabilization by the interstitial (C and N) partitioning to the austenite during the

slow cool. Indirect support for this conclusion was derived from the observation that the martensite in the air cooled steel was twinned (higher carbon) compared to lath martensite (lower carbon) in the oil quenched steel. No twinned martensite was observed in the latter steel.

3. The lower yield strength and superior uniform elongation of the air cooled steel might be a contribution from the austenite transforming to martensite during straining. This would also contribute to the exceptionally high initial strain hardening rates in this steel.

4. The transformed ferrite that formed during air cooling contains a large amount of banded carbonitrides and pinned dislocations and is therefore expected to have a higher yield strength than the retained high temperature ferrite which is relatively carbonitride free.

ACKNOWLEDGMENTS

REFERENCES

1. M. S. Rashid: *SAE Trans.*, 1977, vol. 82, p. 935.
2. S. Hayami and T. Furukawa: *Micro Alloying '75*, Proc. of an Int. Sym. on High Strength Low Alloy Steels held in Washington, DC, in 1975, Union Carbide Corporation, 39th Floor, 270 Park Avenue, New York, NY, pp. 78-87.
3. M. S. Rashid: Sym. Proc. *Formable HSLA and Dual Phase Steels*, A. T. Davenport, ed., TMS-AIME, Warrendale, PA, 1979, pp. 1-24.
4. J. Y. Koo and G. Thomas: *Metall. Trans. A*, 1977, vol. 8A, p. 525.
5. R. G. Davies and C. L. Magee: *Journal of Metals*, November 1979, vol. 31, p. 17.
6. A. R. Marder: Sym. Proc., *Formable HSLA and Dual Phase Steels*, A. T. Davenport, ed., TMS-AIME, Warrendale, PA, 1979, pp. 87-98.
7. J. M. Rigsbee and P. J. VanderArend: Sym. Proc., *Formable HSLA and Dual Phase Steels*, A. T. Davenport, ed., TMS-AIME, Warrendale, PA, 1979, pp. 56-86.
8. P. Ostrom, B. Lonnberg, and I. Lindgren: *Metals Technology*, 1981, vol. 8, p. 81.
9. D. K. Matlock, G. Krauss, L. F. Ramos, and G. S. Huppi: Sym. Proc., *Structure and Properties of Dual Phase Steels*, R. A. Kot and J. W. Morris, eds., TMS-AIME, Warrendale, PA, 1979, pp. 62-90.
10. T. P. Schreiber and A. M. Wims: *Ultramicroscopy*, 1981, vol. 6, p. 323.
11. M. D. Geib, D. K. Matlock, and G. Krauss: *Metall. Trans. A*, 1980, vol. 11A, p. 1683.
12. R. W. K. Honeycombe: *Metall. Trans. A*, 1976, vol. 7A, p. 915.
13. A. R. Marder and G. Krauss: *Trans. ASM*, 1967, vol. 60, pp. 651-60.
14. J. M. Rigsbee: Proc. *ICOMAT-79*, Massachusetts Institute of Technology, Cambridge, MA, 1979, p. 381.
15. G. R. Speich and R. L. Miller: "Mechanical Properties of Ferrite-Martensite Steels," in *Structure and Properties of Dual-Phase Steels*, R. A. Kot and J. W. Morris, eds., TMS-AIME, Warrendale, PA, 1979, pp. 145-82.
16. B. V. N. Rao and M. S. Rashid: "Direct Observations of Deformation Induced Retained Austenite Transformation in GM 980X Dual Phase Steel," General Motors Research Laboratories Publication No. GMR-3576, 1981, Warren, MI 48090.