

The Distribution of Substitutional Alloying Elements during the Bainite Transformation

I. STARK, G.D.W. SMITH, and H.K.D.H. BHADESHIA

The behavior of substitutional alloying elements during and after the growth of upper bainite in Fe-Mn-Si-C and Fe-Mn-Si-C-Mo alloy steels has been examined using an atomic resolution microanalysis technique. From the results obtained, and judging from published data, it is concluded that manganese, nickel, silicon, chromium, and molybdenum do not redistribute during the growth of bainitic ferrite. Their concentrations are found to be uniform both at and in the vicinity of the transformation interface, with no indications of any segregation to the transformation interface during growth. However, prolonged annealing at the isothermal transformation temperature, after the formation of bainite has stopped, eventually stimulates the partitioning of substitutional alloying elements as the system tends toward equilibrium. The results demonstrate the existence of an atomic correspondence between the parent and product phases during transformation, the effect of substitutional alloying additions being manifested *via* a modification of the driving force for transformation.

I. INTRODUCTION

SUBSTITUTIONAL alloying elements can influence the transformation behavior of austenite by several different mechanisms. Of these, the best defined effect is thermodynamic, whereby the alloying elements alter the driving force for transformation from austenite. With all the available thermodynamic data on iron and its alloys, the effect of alloying additions on the driving force for transformation can be estimated to a fair degree of accuracy. However, to apply this information in interpreting the kinetics of transformations requires a prior specification of the conditions which exist at the austenite/ferrite interface during transformation, and this is where there are real difficulties as far as phase changes in steels are concerned. It is generally accepted that there is no long-range redistribution of substitutional alloying elements during the growth of bainitic ferrite in steels.^[1-6] The "bulk" substitutional solute concentration of bainitic ferrite, as measured using electron-probe microanalysis or atom-probe techniques, has long been known to be identical to that of the parent austenite. Growth kinetics are, however, determined largely by the situation at the interface.

In diffusion-controlled growth, it is usual to assume that for binary alloys, the compositions at the transformation interface are determined by a tie line of the phase diagram (see, for example, Reference 7). For alloy steels which are, at the very least, ternary alloys, the situation is more complex. Given the very large difference in the diffusion coefficients of interstitial and sub-

stitutional solutes (X), growth can occur under a variety of conditions, including those in which the equilibrium is somehow constrained. In local paraequilibrium, the Fe/X ratio remains constant everywhere, but subject to that constraint, the carbon redistributes between the phases until its chemical potential is the same in austenite and ferrite at the interface.^[7-11] Since the total concentration of carbon in steels is always small, a constant Fe/X ratio amounts to there being no significant difference in the substitutional alloy concentration of the ferrite and austenite. There is also the negligible-partitioning local equilibrium mode of transformation,^[7,12-15] in which the parent and product phases in the regions away from the interface have almost identical substitutional alloy content, although equilibrium is maintained locally, so that the compositions of the two phases at the interface must, in general, differ, both with respect to carbon and the substitutional solutes. Between these extremes of equilibrium and paraequilibrium, there may exist an infinity of other states in which the chemical potentials of the elements are not equal in the two phases at the interface, although at the same time, none of the elements is completely trapped by the advancing interface.* A situation

*An atomic species is said to be trapped by an advancing interface when its chemical potential increases on transfer into the product phase.^[7,16,17] The species which is trapped may be a solute or a solvent atom. There is no trapping when local equilibrium exists at the transformation interface, whereas the state of paraequilibrium implies the trapping of either X or Fe atoms as the ferrite/austenite interface advances.

like this, in which the alloying elements are partially trapped and partially redistributed during transformation would require some kind of a kinetic constraint to stabilize the motion of the interface and to prevent a collapse toward equilibrium.

In addition to the above complexities, a myriad of generally ill-defined solute/interface interactions have been proposed to significantly influence the kinetics of the bainite reaction. Some of these involve the segregation of substitutional alloying elements to the transformation interface.^[7,18]

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It is evident that high-resolution microanalytical information from regions in the vicinity of the interface can be extremely useful in interpreting the transformation mechanism and kinetics. The present work deals specifically with atom-probe experiments in which the bainite/austenite interface has been analyzed at a high chemical and spatial resolution, in steels where other transformations do not overlap with the formation of bainitic ferrite, and where the reaction occurs at a temperature sufficiently low to avoid significant annealing of the substitutional element distribution during or immediately after bainite growth. Consequently, it has also been possible to identify and study the onset of alloy diffusion during prolonged annealing after transformation.

II. EXPERIMENTAL TECHNIQUES

The high-purity alloys used had the chemical compositions (weight percent) Fe-0.43C-2.2Si-2.8Mn and Fe-0.33C-2.16Si-2.8Mn-0.52Mo. They were homogenized by heat-treating samples in quartz tubes sealed under a partial pressure of pure argon. The homogenization heat treatment involved the holding of samples at 1220 °C for 3 days followed by quenching into brine. To obtain the bainitic microstructure, 1-mm-diameter rods of the alloys were re-austenitized at 1070 °C for 10 minutes before the silica tube was broken and the specimen quenched into a molten salt bath at the appropriate temperature. The tubes were contained in wire baskets, each attached to a long wire handle. This allowed the tubes to be removed from the furnace, broken, and the specimen + basket quenched into the salt bath in approximately 2 seconds. After isothermal transformation to bainite, the samples were quenched into brine. Subsequent optical microscopy and atom-probe analysis of the carbon levels in fully martensitic specimens confirmed that there was no significant decarburization of any of the samples. In all cases, the transformation temperature was at or below 400 °C, and the high silicon concentration ensured that the formation of cementite was severely inhibited. The microstructures obtained thus consisted of just bainitic ferrite and residual austenite (some of which decomposed to martensite on cooling the samples to ambient temperature). The microanalysis experiments were carried out using a VG Scientific FIM100 instrument with the tip temperature set at 90 K. Molybdenum atoms in iron-molybdenum solid solutions image particularly brightly in neon,^[19] and their general distribution could be deduced from the field-ion images. The preferential evaporation effects which arise with silicon steels^[20] were countered by the use of a 20 pct pulse fraction. The samples were prepared for field-ion microscopy (FIM) by necking approximately 0.7-mm-diameter rods in a layer of 25 pct perchloric acid in acetic acid, floated on carbon tetrachloride. Because the polishing process raises the solution temperature above ambient, the voltage was decreased from 20 to 15 V during the first stage, which lasted 5 to 10 minutes. Stable tips were then produced by back electropolishing (18 V) in 2 pct perchloric acid dissolved in 2-butoxyethanol at room temperature. The scatter bars associated with plots of experimental data all correspond to plus and minus one standard deviation.

III. RESULTS AND DISCUSSION

In all of the results reported below, the time at the isothermal transformation temperature was in excess of that required to allow the formation of bainitic ferrite to cease. This is of use in interpreting the terminal carbon concentration of the residual austenite at the stage where the formation of bainite ceases, with respect to the mechanism of transformation.

A. Short Transformation Time

Figure 1 shows the composition profile across a bainitic ferrite/austenite interface in the Fe-Mn-Si-C alloy after isothermal reaction at 358 °C for 2 hours. There is no obvious segregation of carbon, silicon, or manganese at the interface.

Similar conclusions on the distribution of molybdenum at a bainitic ferrite/austenite interface in the Fe-Mn-Si-Mo-C alloy can be drawn from Figure 2, which shows an interphase interface after isothermal transformation for 3 hours at 375 °C. As expected,^[21] the ferrite wedge shows some structure in the field-ion image, whereas the austenite contains only a few isolated bright spots and no other evidence of structure. Austenite tends to image darker because of the easier field evaporation associated with that phase. This difference in imaging character was constantly observed during DC field evaporation between 4.5 and 6 kV, and at no stage was there any suggestion of molybdenum segregation at the interface. The exact position of the interface was more readily discernible during continuous field evaporation than in FIM images.

Molybdenum atoms in iron image very brightly,^[19] direct observation, therefore, is probably the most sensitive method of finding higher than average concentrations of molybdenum at the interface, particularly where the atom-probe detector efficiency is only 20 to 30 pct. The case of desegregation of molybdenum in the present alloy, which has only ≈ 0.3 at. pct Mo, would be very difficult to establish either by observation or atom-probe analysis.

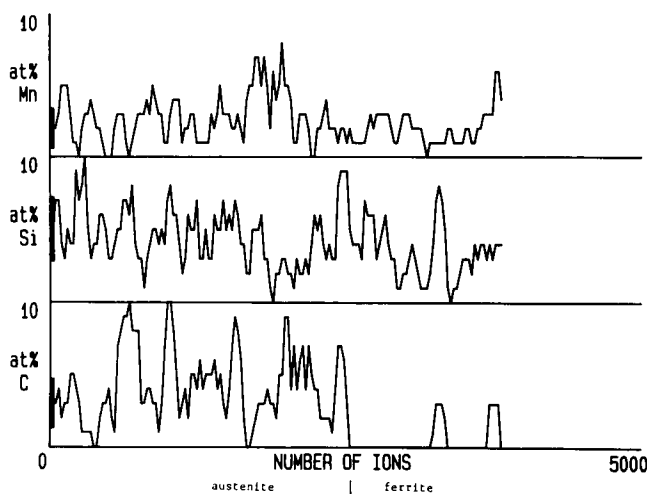


Fig. 1 — Analysis across a bainitic ferrite/austenite interface in an Fe-0.43C-2.24Si-2.82Mn wt pct alloy. The alloy was isothermally transformed at 358 °C for 2 h.

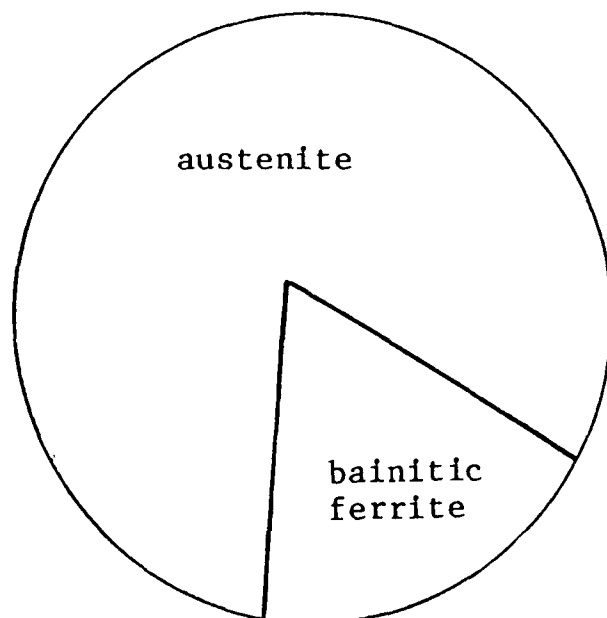
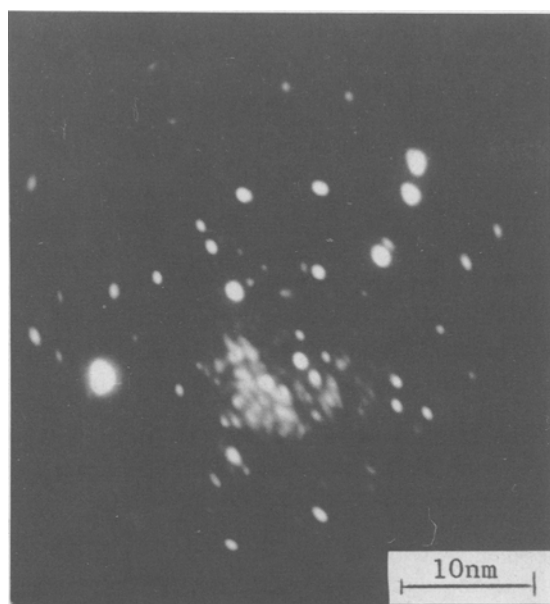


Fig. 2—FIM micrograph showing a bainitic ferrite/austenite interface in an Fe-0.33C-2.16Si-2.8Mn-0.52Mo wt pct alloy after isothermal transformation at 375 °C for 3 h.

Some microanalytical data for the Fe-Mn-Si-Mo-C alloy are shown in Table I; the “bulk” concentrations of substitutional solutes were always found to be the same for both bainitic ferrite and austenite, within the limits of experimental error. The observed carbon concentrations of the residual austenite, at the point where the formation of bainite ceases, are compared against the T_0 , T'_0 , $Ae3'$, and $Ae3''$ phase boundaries in Figure 3 (the diagram was calculated as in Reference 22). The T_0 curve represents the locus of all points where austenite and ferrite of the

same composition have equal free energy.^[23,24] The T'_0 curve allows for 400 J/mol of stored energy in the bainitic ferrite,^[22] to take account of the strain energy due to the invariant-plane strain shape change that accompanies the growth of bainitic ferrite; both the T_0 and T'_0 curves allow for the Zener ordering of carbon atoms in supersaturated ferrite. The $Ae3'$ curve is the paraequilibrium ($\alpha + \gamma$)/ γ phase boundary, and if the free energy of ferrite is increased by the 400 J/mol, then the $Ae3''$ curve is generated. It is evident from Figure 3 that the bainite

Table I. Atom-Probe Analysis of Fe-0.33C-0.52Mo-2.16Si-2.8Mn Weight Percent Alloy with a Microstructure Consisting of a Mixture of Bainitic Ferrite and Austenite*

°C	Minutes	Phase	Carbon		Molybdenum		Silicon		Manganese	
			Pct	SD	Pct	SD	Pct	SD	Pct	SD
410	30	γ	1.1	0.15	0.71	0.12	7.0	0.4	1.9	0.2
410	30	γ	1.4	0.08	0.30	0.04	4.1	0.15	3.0	0.13
410	30	γ	2.3	0.17	0.56	0.08	4.5	0.24	3.1	0.20
410	30	γ	1.2	0.1	0.27	0.05	3.9	0.2	2.6	0.14
410	30	γ	2.0	0.12	0.41	0.05	3.8	0.16	3.0	0.14
410	30	α	0.72	0.05	0.41	0.04	4.9	0.08	2.5	0.10
385	210	γ	2.8	0.19	0.41	0.07	3.8	0.22	2.1	0.17
385	210	γ	2.9	0.54	1.0	0.33	6.7	0.80	2.8	0.53
385	210	γ	4.9	0.60	0.70	0.23	4.7	0.58	3.8	0.54
385	210	α	0.87	0.11	0.39	0.07	4.2	0.23	2.3	0.17
385	210	α	0.08	0.06	0.32	0.12	4.7	0.43	2.9	0.34
385	210	α	0.3	0.05	0.40	0.06	3.8	0.17	3.4	0.16
375	180	γ	2.2	0.07	0.2	0.05	3.4	0.2	2.8	0.2
375	180	γ	2.1	0.3	0.3	0.13	3.8	0.4	2.6	0.36
375	180	α	0.1	0.1	0.5	0.15	3.9	0.4	2.2	0.3
370	30	γ	2.4	0.6	0.64	0.32	6.1	1.0	3.0	0.7
370	30	α	0.64	0.15	0.46	0.13	4.7	0.4	2.2	0.3
370	30	α	0.67	0.11	0.83	0.12	6.0	0.33	2.6	0.22
370	30	α	0.98	0.12	0.66	0.10	6.0	0.3	3.3	0.21

*The concentrations are in atomic percent, and each concentration term is followed by an indication of the standard deviation (SD).

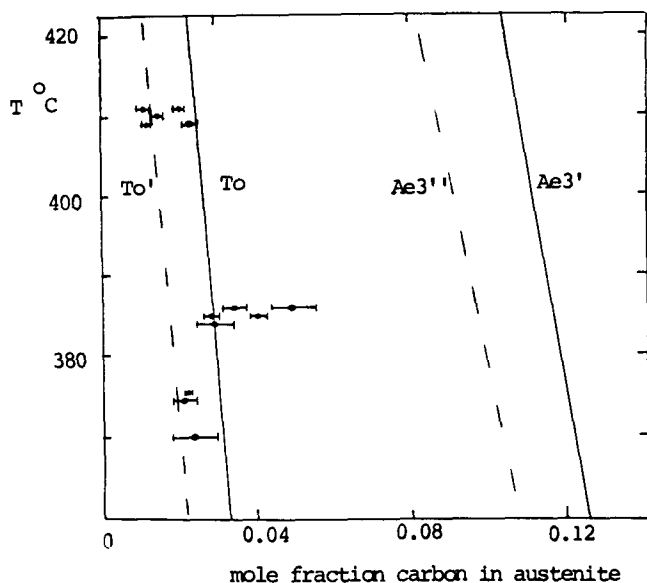


Fig. 3—Calculated phase boundaries for the Fe-0.33C-2.16Si-2.8Mn-0.52Mo wt pct alloy (computed as in Bhadeshia and Edmonds⁽²²⁾). The dashed curves represent the T_0' and $Ae3''$ boundaries, which differ from the T_0 and $Ae3'$ boundaries, respectively, in that they allow for 400 J/mol of stored energy due to transformation. The points represent atom-probe measurements of the carbon concentrations in the austenite which is left untransformed after the completion of the bainite reaction. Note that the exact heat-treatment temperatures are given in Table I; to avoid overlap between adjacent points, some of the data are slightly displaced vertically in the plot presented above. The figure also includes two additional points which are not included in Table I, since these represent austenite carbon concentration measurements done using a lower resolution atom probe. The two points are from samples transformed at 385 °C for 210 min, giving austenite carbon concentrations of 4.0 ± 0.2 and 3.4 ± 0.3 at. pct. The error bars in all cases represent a standard deviation about the point concerned, corresponding to the values of the standard deviations listed in Table I.

reaction stops as the carbon concentration of the residual austenite reaches approximately the T_0 curve, well before the paraequilibrium carbon concentration (given by the $Ae3'$ curve) is achieved. Therefore, the reaction stops before equilibrium or paraequilibrium is attained and is said to exhibit an incomplete reaction phenomenon.^[23,24]

The observations can be explained if it is assumed that the growth of bainitic ferrite is diffusionless, but that unlike martensite, the growth of each platelet of bainite is followed immediately by the rejection of its excess carbon into the residual austenite by diffusion.^[23,24] This is feasible because at the relatively high temperatures associated with the upper bainite transformation, a supersaturated plate of bainite can be decarburized very rapidly.^[24] Hence, as the growth of each plate terminates, the carbon that it rejects enriches the residual austenite so that any further increment of transformation is associated with a lower free energy change. A stage is eventually reached where diffusionless growth becomes thermodynamically impossible as the carbon concentration of the austenite reaches the T_0' curve, and the formation of bainite ceases. This is, of course, what is observed experimentally. Note that the terminal carbon concentration in the austenite is not expected to comply exactly with the T_0' curve, since regions of austenite which are trapped between platelets of bainite can be expected to have a higher carbon concentration,^[3] even though any

austenite must cease to undergo diffusionless transformation to bainite as soon as its carbon concentration reaches the T_0' curve. To summarize, the observations presented in Figure 3 support the hypothesis that the growth of bainite is diffusionless, with the excess carbon being rejected into the residual austenite subsequent to transformation.

B. Prolonged Isothermal Heat Treatment

Specimens of the Fe-Mn-Si-C alloy were isothermally transformed at temperatures between 326 °C and 358 °C for time periods ranging from 17 to 264 hours for detailed atom-probe experiments. For all the samples studied, high manganese levels were consistently found in the close proximity of the interface. The scale of the effect was too small (Figures 4 through 6) to reach an immediate conclusion about whether the observed Mn enrichment is within the interface or in the region of the austenite adjacent to the interface. A further difficulty is that if the interface is not perfectly normal to the probe axis, then the concentrations detected while probing through the interface should, to some degree, average out. The experiments were repeated ten times with similar results being obtained in all cases, as the interface passed through the probed region. The probe hole was initially positioned using FIM imaging. Analyses were carried out with image gas removed, and the position of the interface was considered to be the point where significant carbon levels began to be consistently detected.

It was found that experiments carried out with the image gas present and with the probe hole positioned throughout at the interface failed to detect the Mn enrichment. This is presumably because this particular technique collects simultaneously from both the adjacent phases, leading to an averaging of any composition variation if the enrichment of manganese of one phase is compensated by depletion in the other. This is good evidence to suggest that the changes in manganese concentration, as observed after prolonged annealing at the isothermal transformation temperature, are due to a redistribution of manganese from the ferrite into the austenite, rather than due to any segregation to the interface; a redistribution like this is expected as the system tends toward equilibrium.

The most sensitive method to detect enrichment clearly is to analyze continuously across the interface. In this case, the peak manganese level and the apparent extent of the ferrite depletion layer are both expected to be greatest where the interface/specimen-surface angle is lowest. Because the analysis at any point is an average over 1 to 2 nm, the observed peak concentrations of ≈ 5 to 10 at. pct Mn may be significantly below the actual peak levels.

As already pointed out, the manganese effects, which appear during prolonged isothermal holding, are unlikely to be primarily the result of segregation at the α_b/γ interface, but are probably the first signs of the approach to equilibrium, although some combination of the two phenomena of segregation and redistribution cannot be ruled out. Any segregation to the interface must occur after transformation, since no segregation or redistribution could be detected at the α_b/γ interface for relatively short

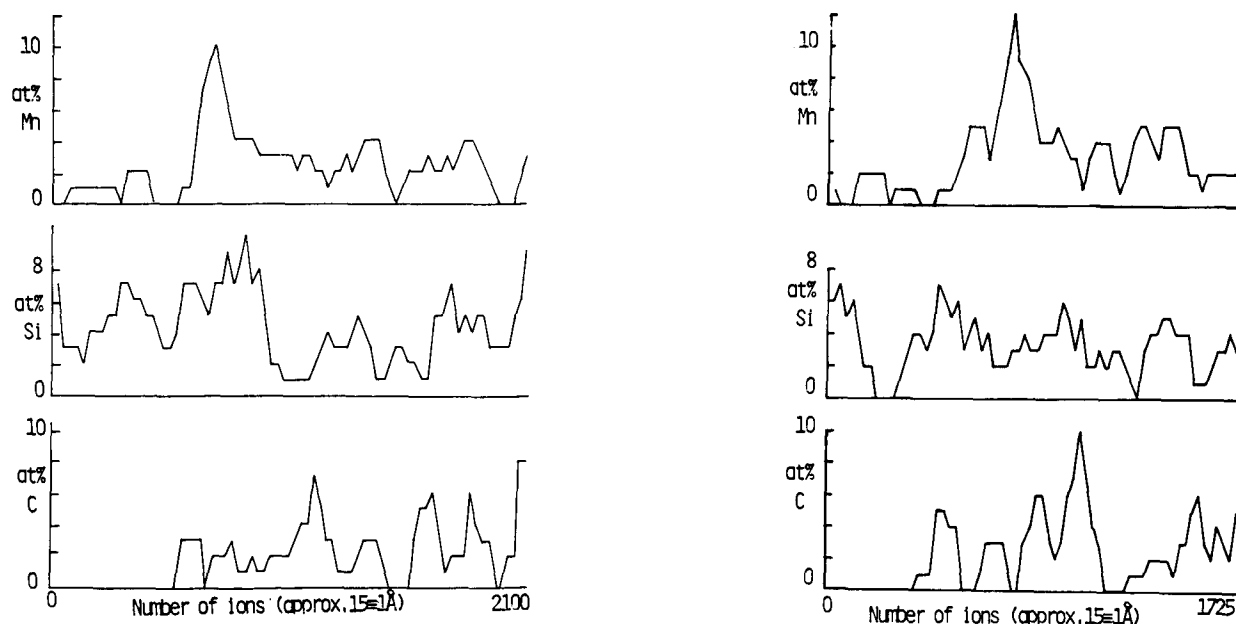
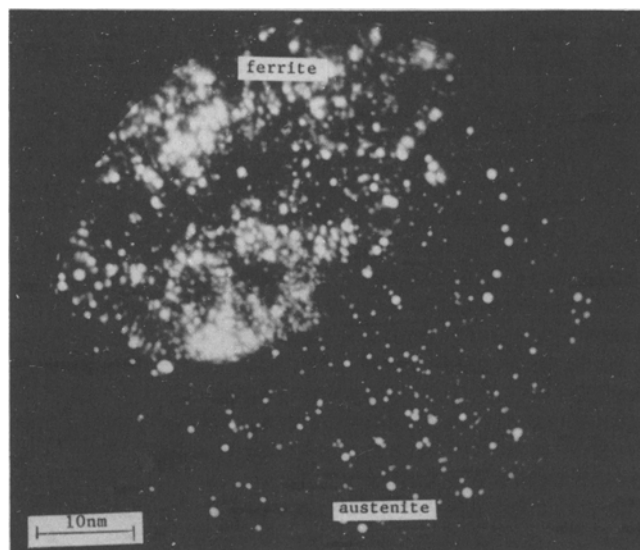


Fig. 4—FIM micrograph showing a bainitic ferrite (light)/austenite (dark) interface in the Fe-0.43C-2.24Si-2.82Mn wt pct alloy after isothermal transformation at 358 °C for 17 h. The composition profiles obtained across the interface are also shown. The position of the interface can be identified approximately as the point where the carbon concentration becomes relatively large. Note the peak in manganese concentration in the austenite at the interface and a corresponding depletion of manganese in the ferrite.

transformation periods, bearing in mind that even these transformation periods were long enough to permit the formation of bainitic ferrite to reach completion. In fact, substantial segregation even after transformation seems unlikely, since the interface concerned is glissile and semicoherent, as indicated by the invariant-plane strain shape change accompanying the growth of bainite.^[22,23,24] Indeed, there was no evidence of carbon enrichment at the interface even after prolonged annealing, consistent with the high coherency of the interface.

The conclusion that the observed manganese concentration changes represent the redistribution of manganese between ferrite and austenite is also supported by the presence of manganese-depleted regions in the ferrite but

not in austenite. The calculated equilibrium compositions (mole fractions) of austenite and ferrite at 415 °C are

	Fe	C	Si	Mn
Ferrite	0.955	0.00007	0.043	0.002
Austenite	0.506	0.204	0.0000004	0.290

The compositions are calculated using thermodynamic modeling based on the THERMOCALC program of the Swedish Royal Institute of Technology and should be regarded as approximate, since the data have been extrapolated to low temperatures. Allowing for the fact that the effective aperture (probe hole) size implies that the analysis at any point is an average over 1 to 2 nm, the observed Mn enrichment in the austenite is consistent

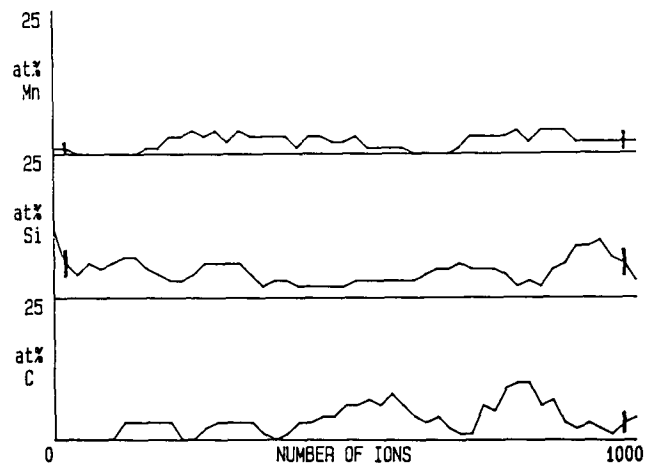
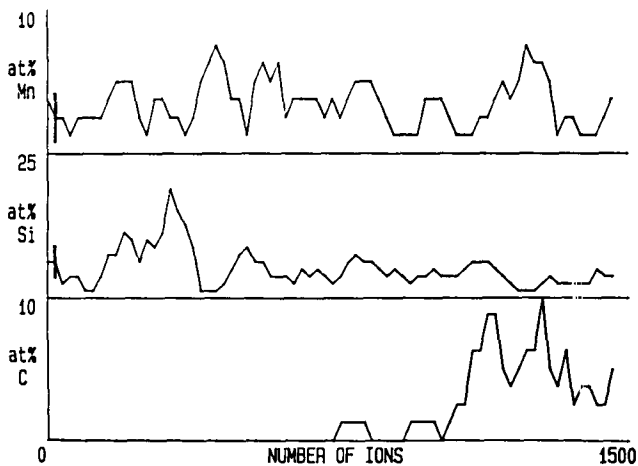
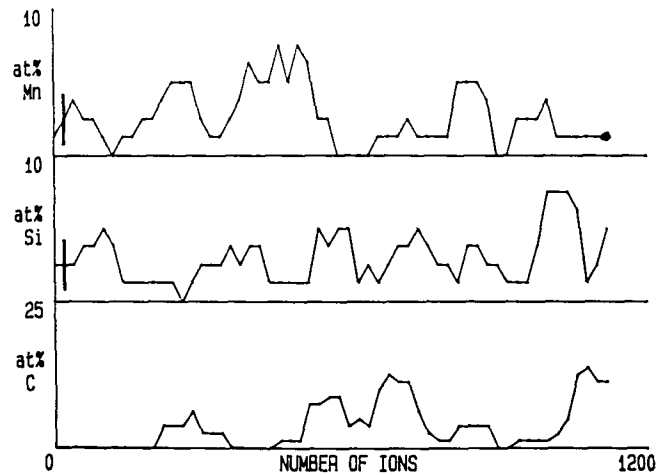
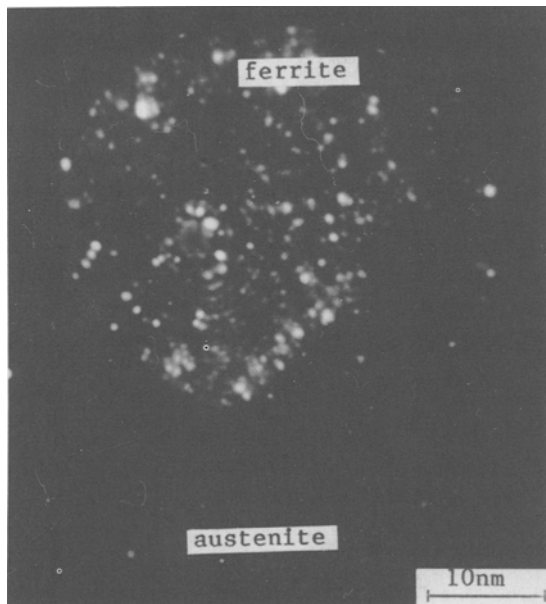


Fig. 5—FIM micrograph showing a bainitic ferrite (light)/austenite (dark) interface in the Fe-0.43C-2.24Si-2.82Mn wt pct alloy after isothermal transformation at 328 °C for 11 days. The composition profiles obtained across the interface are also shown. The position of the interface in each case corresponds to the point where significant levels of carbon begin to be detected.

with the equilibrium composition listed above. The fact that silicon variations are not observed after prolonged annealing could be because of the relatively lower diffusivity of silicon in austenite and because the absolute difference in the equilibrium concentrations of silicon in ferrite and austenite is much smaller.

It is interesting to note that the two effects described above, *i.e.*, the rejection of carbon into the residual austenite immediately after the growth event and the more sluggish redistribution of substitutional alloying additions during prolonged annealing, are both experiments directly analogous to the classical Darken experiment. Diffusion is driven by the need to eliminate chemical potential gradients rather than by the need to homogenize chemically all regions.

C. Summary and Discussion of Published Data

Bach *et al.*^[26] first published atom-probe data on the bainite reaction, data which were interpreted to imply

that there is a redistribution of chromium and molybdenum during the formation of bainite. These results are, however, doubtful, since they indicate an enrichment of Cr and Mo on both sides of the transformation interface. The concentrations were sometimes found to exceed the average concentrations in the alloy used in both phases. Subsequent work by Bhadeshia and Waugh,^[2,3] Stark *et al.*,^[4] and Josefsson and Andren^[6] on steels in which mixtures of just bainitic ferrite and austenite were examined for a variety of steels, established that manganese, silicon, nickel, chromium, and molybdenum do not redistribute at all during the bainite reaction. The present work confirms that Mn, Si, and Mo do not partition during the bainite reaction, even on the finest conceivable scale at the bainitic ferrite/austenite interface. Furthermore, prolonged holding at the isothermal transformation temperature beyond the time period necessary to complete the bainite reaction is found to initiate the process of equilibration. In the case of manganese, this

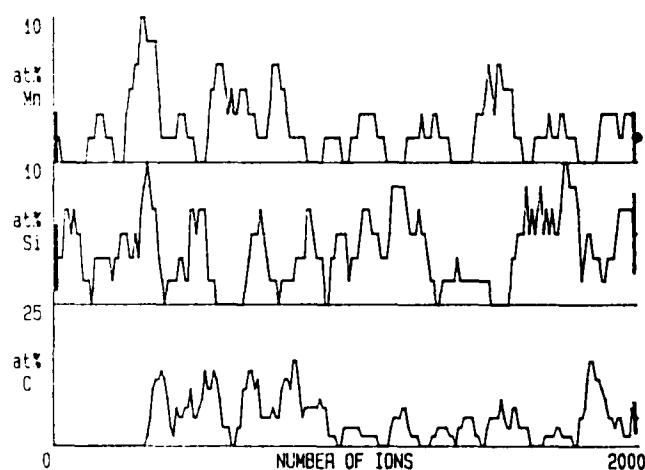
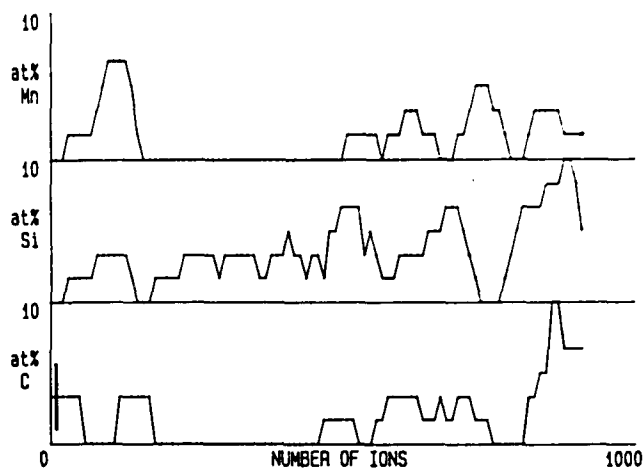
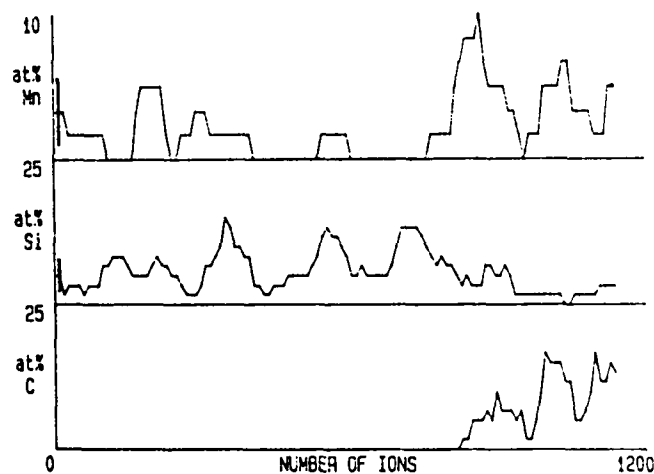
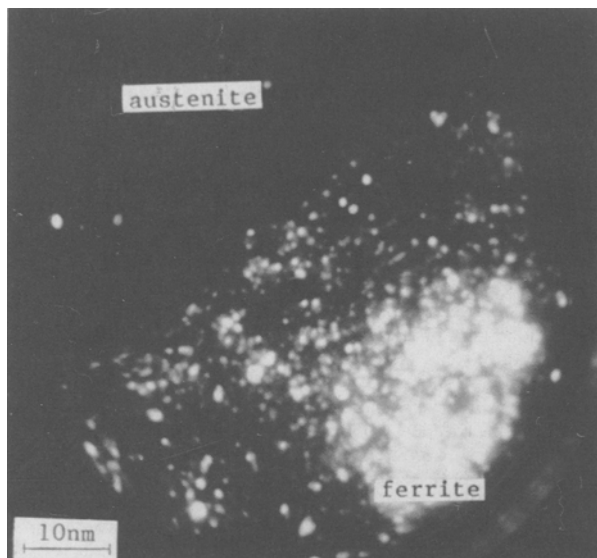


Fig. 6—FIM micrograph showing a bainitic ferrite (light)/austenite (dark) interface in the Fe-0.43C-2.24Si-2.82Mn wt pct alloy after isothermal transformation at 328 °C for 11 days. The composition profiles obtained across the interface are also shown. The position of the interface corresponds to the point where significant levels of carbon begin to be detected.

leads to a redistribution from ferrite into austenite, with the formation of a manganese-depleted zone in the ferrite, and a corresponding manganese-enriched zone in the austenite is not detected in both the ferrite and austenite at the interface. That post-transformation segregation of manganese to the transformation interface does not occur to any significant degree is indicated by the fact that the average composition of the interface and its adjacent regions remains unchanged.

Because of the relatively low transformation temperatures used in the present work, the diffusional processes which allow the manganese to partition are found to be very sluggish. This is an important observation, since it provides further evidence for a lack of atomic mobility at the temperatures and time scales typically associated with the formation of bainite. The evidence supports a displacive transformation mechanism in which the iron and substitutional atoms are configurationally frozen during transformation. On the other hand, in a diffusional reaction, the reconstructive diffusion necessary to accomplish the lattice change^[7] should, at the same time,

provide an opportunity for the simultaneous partitioning of substitutional solutes.

These observations are also consistent with the fact that an invariant-plane strain shape change accompanies the growth of bainitic ferrite;^[22-25] such a shape change is generally taken to imply the existence of an atomic correspondence between the parent and product phases, at least as far as the substitutional solute and iron atoms are concerned. Hence, diffusion of substitutional species is not expected during transformation.

Since the atomic correspondence discussed above must exist across *any* interface between a plate of bainitic ferrite and its parent austenite,^[23] the present results and observations are valid for any interface orientation.

Finally, it should be noted that the segregation of molybdenum to austenite/ferrite interfaces for high-temperature transformations has been studied elsewhere,^[27,28,29] but the ferrite microstructures involved were not bainitic. The claimed segregation was also not statistically significant with respect to the reported noise levels.

IV. CONCLUSIONS

Our experimental data, when combined with published work on the role of substitutional solutes during the bainite reaction, demonstrate that in a variety of steels, none of the elements, Mn, Si, Ni, Mo, or Cr, partitions during transformation, even on the finest conceivable scale. Nor do these elements segregate to the transformation interface. The results are consistent with the observed features of the bainite reaction, such as the shape deformation accompanying the growth of bainitic ferrite and with the existence of an atomic correspondence between the parent and product phases during transformation.

Given an opportunity, the substitutional solutes do eventually begin to redistribute so as to lower the free energy of the system, albeit at a very sluggish pace. The fact that such redistribution is not at all evident during the bainite reaction is an indication of the absence of any reconstructive diffusion during the growth of bainite and emphasizes the displacive character of the bainite reaction.

Further results have been obtained confirming the incomplete reaction phenomenon which indicates that the growth of bainitic ferrite is diffusionless, with any excess carbon being rejected into the residual austenite subsequent to transformation.

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