Austenite films in bainitic microstructures

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Bainitic microstructures in which fine platelets of ferrite are intimately mixed with films of austenite are known to exhibit good combinations of strength and toughness. It is found that the thickness of these austenite films can be estimated by assuming that the carbon diffusion field around an existing plate of ferrite prevents the close approach of another parallel plate. This is because the regions of austenite with the highest carbon concentration are unable to transform to bainite. MST/3052

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Introduction

It is now well established that substantial quantities of austenite can be retained in the microstructure when silicon rich steels are transformed to bainite.¹⁻⁴ This is because the silicon and other elements, which have low solubility in cementite, retard its precipitation. The carbon that is partitioned into the austenite as a consequence of the formation of bainitic ferrite therefore remains in the austenite, stabilising it to ambient temperature.

A microstructure such as this, in which fine plates of bainitic ferrite are separated by films of austenite, is in many respects ideal from the viewpoint of mechanical properties. The fine scale of the ferrite plates is conducive to good toughness, as is the absence of carbides (which have the potential of nucleating cleavage cracks or voids). The ductile austenite films which are mixed intimately with the ferrite are also believed to be beneficial to toughness.

Many features of bainitic microstructures in silicon rich steels can be calculated theoretically. These include the volume fractions of bainitic ferrite and austenite as a function of steel composition and transformation temperature, and the chemical compositions of the austenite and bainitic ferrite. However, the factors controlling the thickness of the austenite films are not yet established, and the films themselves have never been characterised quantitatively. The purpose of the present work was to study a series of experimental silicon rich steels to develop and verify a theory for the factors controlling the thickness of the austenite films.

Experimental method

The chemical compositions of the experimental alloys are given in Table 1. The alloys were made as vacuum melts in the Swinden Laboratories of British Steel. The as cast ingots were forged and rolled down to plates 16 mm in thickness and 150 mm in width. The alloys all contain sufficient silicon (about 2 wt-%) to prevent the precipitation of cementite during transformation to bainite, and about 2 wt-% manganese for the purposes of hardenability. The carbon, nickel, and chromium concentrations are significantly different to obtain corresponding variations in the stability of the austenite.

The steels were first machined into 10 mm dia. rods, sealed in quartz tubes containing a partial argon atmosphere, and then given a homogenisation heat treatment at 1300°C for three days, followed by furnace cooling to room temperature. Samples were then machined from the homogenised specimens into cylindrical shapes, each of 8 mm dia. and 12 mm length. To ensure rapid cooling, each cylinder

was drilled to give a 3 mm dia. longitudinal hole. The specimens were then nickel plated using a two stage procedure, striking and plating. The striking process used a solution consisting of 250 g nickel sulphate and 27 mL concentrated sulphuric acid in 1 L distilled water, at 50°C for 3 min and a current density of 7.7 mA mm⁻². The plating was carried out at 50°C for 15 min with a current density of 0.4 mA mm⁻² in an electrolyte containing 140 g nickel sulphate, 15 g ammonium chloride, and 20 g boric acid in 1 L distilled water.

DILATOMETRY

Isothermal transformation was carried out in a Thermecmastor thermomechanical simulator capable of monitoring the strain, load, time, and temperature during the course of transformation. The machine is equipped with an environmental chamber which can be evacuated using a rotary and oil diffusion pump, or which can be filled with inert gas. The heat source is a radio frequency coil, and the temperature was measured using a Pt/Pt–10%Rh thermocouple spot welded to the sample. The temperature variation along the length of the sample was checked to be within 4–5 K.

The samples were austenitised by heating to 1000° C for 10 min in the vacuum chamber, then quenched using a nitrogen or helium jet to a variety of isothermal temperatures below the bainite start temperature B_s but above the martensite start temperature M_s . The diameter change during transformation was recorded to relate this to the volume change on transformation and to estimate the carbon concentration of the austenite at the point at which isothermal transformation stops, using a method described elsewhere in detail.⁵

OPTICAL AND TRANSMISSION ELECTRON MICROSCOPY

The specimens were prepared for optical microscopy by mounting in conductive Bakelite, grinding using 120 grit silicon carbide paper, and finally, polishing down to 1 μ m diamond grit. They were etched using 2% nital.

Thin foils for transmission electron microscopy (TEM) were prepared from 0.3 mm thick discs machined from

Table 1 Composition of alloys used (wt-%)

Alloy	С	Si	Mn	Cr	Ni
A	0.27	1.98	2.18	1.9	
В	0.27	2.01	2.16		2.07
С	0.46	2.10	2.15		
D	0.44	2.13	2.14	0.2	
E	0.1	1.77	2.12		2.0
Н	0.095	1.63	1.99	1.97	
К	0.26	1.85	2.10		
V	0.26	1.93	2.04	1.02	



1 Linear intercept L_{T} obtained when test plane is restricted to rotation about one of long edges of plate, and test line is restricted to orientation normal to longitudinal direction of plate in test plane

8 mm dia. rods. The discs were ground down to $6-80\,\mu\text{m}$ thickness by abrasion using silicon carbide coated grinding paper, and electropolished in a twin jet polishing unit containing an electrolyte of 5% perchloric acid, 25% glycerol, and 70% ethyl alcohol. The polishing potential was 55 V at a current of 20-30 mA, the electrolyte temperature being maintained in the range -5 to -10° C. The thin foils were examined using a Philips EM-400ST transmission electron microscope operating at 120 kV. A considerable amount of TEM was performed to make quantitative measurements of the ferrite plate and austenite film sizes. This included electron diffraction and dark field imaging to identify all the phases conclusively. However, much of the work is not reported in the present paper because such microstructures have been investigated qualitatively on many previous occasions.^{2–4} The microstructures illustrated in the present paper are only required for a general overview.

STEREOLOGICAL MEASUREMENTS

The austenite film thicknesses were measured directly from TEM at magnifications ranging from $\times 17000$ to $\times 36000$. The shortest distance perpendicular to the longitudinal dimension of the austenite film was measured in each instance. The same procedure was used to measure the apparent thickness of the bainitic ferrite subunits. Approximately 30–100 measurements were made for each heat treatment.

Some stereological corrections are necessary since the measurements were made on random sections. For a square plate of thickness t and sides $a \times a$, the mean linear intercept obtained by projecting random test lines on to random two-dimensional sections is given by⁶

when $a \gg t$. However, for the present work it was much more convenient to measure the mean intercept \overline{L}_N , which is obtained by orienting the test line in a direction normal to the longitudinal dimension of the film or plate. The effect of this is shown in Fig. 1; it is as though the plane of observation always contains a long edge of the plate, with the additional condition that the test line is normal to this long edge. The perimeter P_A per unit area (Fig. 1) is given by

$$P_{\rm A} = \frac{(t+a)}{at} \approx \frac{1}{t} \qquad \dots \qquad \dots \qquad \dots \qquad \dots \qquad \dots \qquad (2)$$

Note that the perimeter is taken to be a+t rather than 2(a+t) because the interface is shared between adjacent

grains. Using the standard stereological relationship⁷

it follows that

Comparison with equation (1) shows, as might be expected, that the linear intercept as measured here is closer to the true thickness than it would be if the test line were completely randomly oriented. All the ferrite and austenite thickness data reported here have therefore been corrected by the factor $\frac{\pi}{2}$.

General microstructure

There are two essential morphologies of austenite in silicon rich steels which are transformed to bainite. There are films of austenite between the individual platelets (subunits) of bainitic ferrite, and the coarser, more equiaxed blocks of austenite between non-parallel sheaves of bainite.^{3,4} The blocks of austenite are easily visible in the optical microstructure, as shown in Fig. 2*a*. The sample illustrated was isothermally transformed at 300°C until the formation of bainite stopped, so that the fraction of bainite shown is a limiting fraction. Since the limiting fraction is smaller at higher temperatures (until it becomes zero at B_s), the blocks of austenite become larger when the isothermal transformation is at a higher temperature (Fig. 2*b*).

The film austenite can only be resolved using TEM, as shown in Fig. 3a. The films of austenite are dispersed between the subunits of bainitic ferrite. Figure 4 shows that the films of austenite clearly become finer as the transformation temperature is reduced, or as the carbon concentration is reduced.

Figure 5a shows that the distinction between blocky austenite and film austenite becomes less clear when the blocks of austenite occur between parallel sheaves. However the thickness of the blocks is clearly much greater than the film thickness and the blocks extend to greater lengths, so there was usually no difficulty in distinguishing between interplate films of austenite and intersheaf blocks of austenite. However, the extent of transformation in the lowest carbon alloys can be so large as to make the individual sheaf indistinct. For example, Fig. 5b shows the low carbon alloy E transformed at 380°C, where there are no large regions of austenite between different sheaves, although there is obviously a bimodal distribution of austenite thickness. The measurements of austenite film thickness in those instances were confined to the fine films which truly represent the films between the ferrite subunits.

Mechanism of transformation

The analysis of experimental film thickness measurements requires a knowledge of the mechanism of transformation. There is evidence to suggest that although the plates of bainite grow by a displacive, diffusionless transformation mechanism, any excess carbon in the ferrite is soon afterwards partitioned into the residual austenite.⁸ The austenite thus progressively enriches in carbon, until a point is reached at which it becomes thermodynamically impossible for further transformation to occur via this mechanism.

It is useful to define a number of thermodynamic concepts consistent with this mechanism of bainite growth.



a heat treated at 1000 C for 60 s and 300 C for 2000 s before gas quenching to room temperature; b heat treatment as for a, but isothermally transformed at 460 C (coarser blocks of austenite can be seen)

2 Optical micrographs of alloy C, showing mixture of bainitic ferrite and austenite, (white etching phase): darker etching regions consist of mixture of fine bainitic ferrite platelets separated by films of austenite, which can not be resolved optically

The T_0 curve represents the locus of all points on a plot of temperature versus carbon concentration, where austenite and ferrite of the same chemical composition also have identical free energies. Hence, austenite with a carbon concentration in excess of that defined by the T_0 curve cannot, even in principle, transform without diffusion. The

3 a alloy C isothermally transformed at 350°C, showing films of austenite and b corresponding austenite dark field image

growth of bainite is accompanied by a change in shape which is characterised as an invariant-plane strain with a large shear component. The strains caused in the austenite as it accommodates this shape deformation are associated with an estimated 400 J mol⁻¹ of strain energy.⁸ The T_0 curve modified to account for this strain energy is called the T'_0 curve.

An alternative possibility is that the transformation mechanism is diffusionless only for the iron and substitutional

a alloy C showing films of austenite between subunits of bainitic ferrite, in sample transformed isothermally at 400°C; *b* as for *a*, but isothermally transformed at 300°C; *c* lower carbon alloy E transformed isothermally at 400°C

4 Transmission electron micrographs showing films of austenite

5 a alloy C isothermally transformed at 300°C, showing blocks of austenite between parallel sheaves of bainite (latter also contains films of austenite) and b alloy E, isothermally transformed at 380°C (distinction of individual sheaf is unclear)

a Ae'₃ condition; b T₀ condition; c T'₀ condition (note magnified scale)
6 Measured carbon concentration of austenite at point where transformation to bainite stops versus calculated concentration for various conditions

solutes. Interstitial carbon may partition to such an extent that the bainitic ferrite is at no stage of its existence supersaturated with carbon. If strain energy is neglected, then the austenite can in these circumstances continue to transform until its carbon concentration reaches the Ae'_3 curve on the temperature–carbon concentration plot. The

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 Ae'_3 curve represents the paraequilibrium $\alpha + \gamma/\gamma$ phase boundary.

To summarise, a completely diffusionless transformation mechanism would be limited by the T'_0 curve, whereas one in which only carbon diffused during transformation would be expected to be limited by the Ae'_3 paraequilibrium phase boundary. As stated above there is accumulated evidence⁸⁻¹⁰ that the transformation to bainite stops when the carbon concentration of the residual austenite reaches the T'_0 boundary. Nevertheless, it was considered useful to verify these principles for the steels considered in the present work, particularly since the heat treatments were all carried out in a thermomechanical simulator so that there was no extra expense in collecting the necessary data.

Isothermal transformation experiments were carried out over a temperature range between $B_{\rm S}$ and $M_{\rm S}$, the exact temperature depending on the alloy concerned. The transformation strain was monitored until reaction ceased. This strain can then be converted into a volume fraction of bainitic ferrite and the fraction can in turn be used to calculate the carbon concentration of the residual austenite at the point at which the reaction ceased. The detailed procedure has been described previously.^{5,9} Figure 6 shows three diagrams comparing the measured austenite carbon concentration against the concentrations expected if the formation of bainite ceases when the austenite carbon concentration reaches that given by the Ae'_3 , T_0 , or T'_0 conditions described above. These latter concentrations can be calculated using well established thermodynamic methods.¹¹ Figure 6a is interesting in two respects. First, it is obvious that the formation of bainitic ferrite ceases well before the austenite achieves the paraequilibrium carbon concentration. Second, the extent of the discrepancy increases as the transformation temperature is reduced (i.e. at larger austenite carbon levels). Both of these results are expected, the latter because the T'_0 curve has a steeper slope on the phase diagram when compared with the Ae'_3 curve.11 The agreement between the experimental data and the calculations improves considerably when the comparison is made against the T_0 concentrations (Fig. 6b), but there is nevertheless an overestimation of the experimental data. The best agreement is clearly when the comparison is against the T'_0 concentrations (Fig. 6c), which are based on diffusionless transformation including a strain energy term, as described above.

These results are useful in that they confirm that for the alloys of interest in the present work, it is reasonable to assume that the plates of bainitic ferrite form initially without diffusion, but that the excess carbon in the ferrite partitions soon after into the residual austenite. The value of stored energy used in the T'_0 calculations, at 400 J mol⁻¹ (Ref. 9) also seems reasonable.

Figure 7 shows the measured austenite film thickness as a function of the measured bainite subunit thickness. It might be expected that a thicker ferrite plate would lead to a thicker austenite film since the amount of carbon partitioned is then larger. Whereas a trend of this type can be seen in Fig. 7, the correlation is poor because (as discussed below) certain factors which depend on the alloy chemistry have not been taken into account. An earlier assumption^{3,4} that the ratio of film/plate thickness should be approximately constant is clearly not justified.

Since the films of austenite are isolated by the adjacent platelets of ferrite, their thickness should not change as transformation progresses. Partial transformation experiments in which the samples were held at temperature for only 150 s, were conducted for alloy C at 300, 350, and 400 °C. The results are plotted together with the data from the fully transformed samples in Fig. 8. The two sets of data are essentially identical, showing that the thickness of the film does not change during the course of the transformation.

7 Experimentally determined austenite film thickness as function of thickness of adjacent ferrite plate, for variety of steels and transformation temperatures: numbers given in parentheses represent carbon concentrations (in wt-%) of alloys concerned – best fit line is determined by data from steels except steels E and H which both contain 0.1 wt-% carbon

Calculation of austenite film thickness

The method used to calculate the thickness of the austenite films is based on the hypothesis that transformation to bainite can only occur in regions of austenite where $x_{\gamma} \leq x_{T_0}$, where x_{γ} is the carbon concentration in the austenite and $x_{T_0'}$ is the carbon concentration corresponding to the T_0' curve for the temperature of interest. As a subunit of bainitic ferrite forms, it partitions its excess carbon into the residual austenite. This creates a carbon diffusion field around the subunit. Another parallel subunit (of the same sheaf) which forms subsequently, can only approach the original subunit to a point where $x_{\gamma} \leq x_{T_0'}$. This is illustrated schematically in Fig. 9. The method assumes that the interval between subunit formation is larger than the time required to decarburise each subunit. The validity of this

Austenite Film Thickness / µm

8 Measured thickness of films of austenite in partial (150 s) and fully transformed specimens for alloy C at temperatures of 300, 350, and 400°C (sets of data are essentially identical)

assumption can only be justified by testing the proposed model for the austenite thickness.

The problem therefore becomes a calculation of the carbon concentration profile normal to the bainitic ferrite subunit habit plane, at the point at which the subunit is decarburised to its paraequilibrium carbon concentration. This should in principle be done using a finite difference method,¹² but an approximate analytical solution is used instead. This is because of the simplicity of the analytical method which also can give greater insight into the physical principles involved. The concentration profile when the excess carbon has all partitioned into the adjacent austenite is therefore given by¹³

where z is a coordinate defined normal to the habit plane, $x^{\gamma \alpha}$ is the paraequilibrium carbon concentration in the austenite, t_d is the time to decarburise the ferrite plate, and \overline{D} is the weighted average diffusivity of carbon in the austenite^{14–16}

The function $D^{\gamma}\{x, T\}$ adopted in the present study is based on the theory of Siller and McLellan,¹⁷ and Bhadeshia.¹⁸ This relationship can be integrated to give

where w_{α} is the thickness of the ferrite plate.

The carbon profiles for alloy A, at the instant of completion of decarburisation are plotted in Fig. 10*a*, for five different temperatures with $x_{T'_0}$ marked in each case. It is clear that the austenite film thickness should increase with temperature. The effect of carbon is illustrated in Fig. 10*b*. An increase in the average carbon concentration, at constant temperature, also leads to an increase in film thickness.

The complete assessment of all measurements is presented in Fig. 11, which shows that the trends in the variation of austenite film thickness as a function of the transformation temperature and alloy chemistry are well predicted, although the experimental data are systematically underestimated. One possibility for the underestimation is the assumption that the carbon diffusion field of a subunit is that which is established immediately after the excess

10 *a* carbon concentration variation in austenite (alloy A) as function of transformation temperature, together with corresponding values of x_{T_0} for each temperature and *b* carbon concentration variation in austenite (alloys A, C, and E) as function of alloy chemistry for particular transformation temperatures (400°C), together with corresponding values of x_{T_0}

carbon has partitioned into the residual austenite. The austenite is clearly not homogeneous at this stage, and diffusion in the austenite will tend to level out the concentration gradients, making the approach distance of the next subunit larger. This is investigated qualitatively below. A fully quantitative study is not yet possible since that would require a knowledge of the time interval between the formation of adjacent subunits.

FINITE DIFFERENCE ANALYSIS

The homogenisation of carbon in the austenite can be studied using a standard finite difference method discussed by Crank.¹⁹ The diffusion process is normal to the α/γ interface, and is symmetrical about the centreline of the ferrite plate, so that only one side of the plate needs to be considered.

For the purpose of numerical analysis, the austenite region was divided into a number of slices n with

11 Comparison of measured and calculated austenite film thicknesses for various alloys

where w_7^s is the thickness of a slice of austenite. Larger values of *n* lead to greater accuracy at the expense of computing time, so *n* was chosen to be between 3000 and 10 000. A check was made to ensure that there was no substantive change in the results when the value of *n* was increased. The initial concentration profile in the austenite (following the decarburisation of ferrite) was set using equation (5). The time interval *t* between successive calculations of the concentration distribution is given by

where r is a grid parameter in the finite difference method, which can be set to a smaller value for higher accuracy; the value of r was fixed by ensuring that the results become insensitive to the choice.

The explicit finite difference formula is

where the normalised concentration $x'_j = x_j/\bar{x}$, *i* is the slice number, and *j* represents the successive time rows of the grid. The first and last slices of each time row follow the relationships

$$x'_{\gamma}(1,j) = x'_{\gamma}(1,j-1) + r[x'_{\gamma}(2,j-1) - x'_{\gamma}(1,j-1)]$$

$$x'_{\gamma}(i_{1}, j) = x'_{\gamma}(i_{1}, j-1) + r[x'_{\gamma}(i_{1}-1, j-1)]$$

$$-2x'_{y}(i_{1}, j-1) + x'_{y}(i_{1}-1, j-1)] \quad . \quad . \quad (12)$$

where i_1 is the number of the last slice. Precautions were taken to ensure that mass was conserved throughout the process.

The method can be used to analyse how the carbon homogenises in the austenite after the plate of bainitic ferrite has decarburised. Figure 12 shows the calculated profiles as a function of time for a number of transformation temperatures (alloy A). The profiles obviously change faster at the higher temperatures, because of the correspondingly larger diffusion coefficients involved. Examination of the x_{T_0} condition shows that the calculated austenite film thickness should, as expected, increase. However, the change is not very large, for what are believed to be realistic time intervals, because in general, $x_{T_0} \ll x^{\gamma z}$.

Summary and conclusions

It appears that the thickness of those austenite films which lie between parallel bainitic ferrite subunits is controlled by the T'_0 condition. As a supersaturated bainite plate grows and partitions its excess carbon into the surrounding austenite, an adjacent carbon enriched austenite layer is created in which the carbon concentration is greater than that which would permit diffusionless transformation. It is this layer which is retained as the thin film of austenite. The general trend in the thickness of the austenite films, as a function of the transformation temperature and alloy composition, can be predicted using a simple analytical model. The model, however, underestimates the thickness because it contains nothing about the time interval between the growth of successive, adjacent subunits.

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