SEGREGATION, OXYGEN CONTENT AND THE TRANSFORMATION

START TEMPERATURE IN STEEL WELD DEPOSITS

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Abstract

The high cooling rates associated with most arc-welding processes causes non-equilibrium solidification so that the final weld is compositionally heterogeneous. The effect of such segregation on the temperature at which the transformation from austenite to ferrite first occurs on cooling from the austenite phase field (i.e., T_h), is investigated both experimentally and theoretically for reheated Fe-C-Mn-Si weld deposits and pure alloys. The role of oxygen concentration in influencing the transformation temperatures is also investigated. It is found that the effect of alloy segregation is to elevate T_h , to an extent depending on the average alloy concentration. For homogeneous alloys, the agreement between experiment and theory for T_h is reasonable, the former being a little higher than expected, but it is demonstrated that the discrepancy has only a small effect on calculations of weld microstructure. It is found that the oxygen content has no noticeable effect on the transformation start temperature of allotriomorphic ferrite in welds at the levels studied (≈ 200 ppm).

Introduction

The microstructure of the fusion zone of a weld, which is not influenced by any subsequent heat-treatment (for example, by the deposition of further passes) is called the primary microstructure. A major step in the evolution of the primary microstructure occurs during the continuous cooling of the weld from the austenite phase field, and is a complex process, which may be summarised as follows.

During cooling of a low-alloy steel weld deposit, the austenite $\langle \gamma \rangle$ first begins to decompose by a diffusional transformation mechanism to allotriomorphic ferrite (α) at a temperature T_h which depends on the cooling rate, alloy composition and the austenite grain size and shape. In order to avoid cracking and to optimise toughness, the alloying of welds with respect to both substitutional and interstitial elements is low. The rate of α growth is therefore very high, and the original γ grain boundaries rapidly become decorated with fairly uniform layer of allotriomorphic ferrite. This ferrite continues to thicken in a direction normal to the γ grain boundaries, until at some temperature T_l , diffusional transformations become relatively sluggish and the austenite then transforms by a displacive mechanism to Widmanstätten ferrite, acicular ferrite or martensite/degenerate pearlite.

Considerable work has recently been done on the prediction of the primary microstructure using detailed phase transformation theory (1-4), and this has met with reasonable success; the method is now being used commercially in the design of welding consumables and procedures. A small but significant part of the calculation requires the temperature T_h which is calculated by first computing (5) an isothermal transformation diagram (as a function of the alloy chemistry). This gives, for a particular transformation, a plot of the incubation time τ_i required to obtain a detectable degree of transformation after quenching from the γ phase field to an isothermal transformation temperature T_i . Of course, transformations during welding occur over a range of temperatures, and this is taken into account by treating the continuous cooling curve as a series of isothermal transformations, so that if the specimen spends a time Δt_i at a temperature T_i , then transformation begins when:

$$\sum_{i} \Delta t_{i} / \tau_{i} = 1$$
(1)

When the sum reaches a value of unity, $T_i=T_h$, when τ_i are for allotriomorphic ferrite (Ref.6 includes a detailed discussion of eq.1). Equation 1 is essentially an *additive reaction rule* and works approximately in converting an isothermal transformation curve into a continuous cooling curve, at least for the beginning of transformation.

During solidification, the cooling rate is sufficiently high to cause considerable chemical segregation. The austenite, when it begins to transform, is therefore far from homogeneous, and this must in turn influence its transformation kinetics. Gretoft *et al.* (7) took this into account by calculating the liquid/delta-ferrite partitioning coefficients of substitutional alloying elements, and hence obtaining the composition of the *solute-depleted* regions of a weld. A separate isothermal transformation diagram was then calculated for the solute-depleted regions, and since austenite transforms more rapidly in these regions, it was used to calculate T_h , which turned out to be higher than that expected for a homogeneous alloy. This also means that the volume fraction of allotriomorphic ferrite obtained is higher for a heterogeneous weld.

Although the procedure discussed above works well, it was decided to investigate further the factors influencing T_h for three reasons:

- (a) It is now necessary to get very accurate predictions of the allotriomorphic ferrite content of a weld; too large a volume fraction of this phase is known to be detrimental to the toughness of a weld, acicular ferrite being better in this respect. However, it is possible to obtain virtually 100% acicular ferrite (given that the cost, strength etc. can be tolerated), but the microstructure then does not give optimum toughness (8). In general, a microstructure containing a small amount of allotriomorphic ferrite with the rest being acicular ferrite is known to give the best toughness. The reason for this is not clear, but could be related to the fact that as the acicular ferrite content increases, so does the yield strength and the level of hard microphases (martensite, degenerate pearlite); also, undecorated prior austenite grain boundaries may be more susceptible to trace impurity element embrittlement.
- (b) It has been suggested (9) that the presence of oxide rich inclusions, for oxygen levels between 320 and 600 ppm, in a weld raises T_h , even though α nucleates at austenite grain boundaries, because the γ grain size is influenced by inclusion content. However, the issue is confused because recent work, at oxygen levels between 220 and 420 ppm, suggests that the columnar γ grains of weld deposits are not pinned by inclusions (10), because they evolve from δ -ferrite, and the driving force for the δ - γ transformation increases indefinitely with undercooling below the equilibrium transformation temperature. Inclusions may however have a pinning effect on grain growth in reheated weld deposits; the driving force for grain growth is approximately independent of temperature and in the present context is rather small compared with that for phase transformations. Furthermore, the original work which claimed a significant effect of inclusions on T_h was based on a doubtful interpretation of length versus temperature dilatometric curves, which were significantly non-linear before transformation; these experiments also did not take any account of surface effects and other details to be discussed later.
- (c) It should be interesting to compare T_h for as-deposited welds with welds which have been homogenised to remove compositional variations.

The purpose of the present work was therefore to study the effect of oxygen concentration and of alloying element segregation on the temperature at which austenite first transforms to allotriomorphic ferrite. It was hoped to compare transformation start temperatures obtained from three submerged arc weld deposits with three corresponding high-purity wrought alloys and calculated data; the pure alloys serve to check theory and any special effects that occur in welds as a direct consequence of impurities such as oxide inclusions. The present work is a part of a programme of research aimed at the accurate calculation of weld microstructure using phase transformation theory.

Experimental Procedures

Dilatometric measurements were carried out using a high-speed Theta Industries dilatometer (which has a RF furnace of virtually zero thermal mass, allowing rapid changes in specimen temperature), the temperature being recorded with a Pt/Pt-10wt.%Rh thermocouple attached directly to the specimen. The dilatometer length transducer was calibrated using a pure platinum specimen of known thermal expansivity; the dilatometer was programmed to give a constant cooling rate during any given experiment; the cooling rates were varied to span a wide variety of welding conditions. Some of the very rapid cooling rates were obtained using a helium gas quenching facility available. Austenitisation was carried out at 1000°C for 10 min with the specimen chamber flooded with helium.

Dilatometer specimens were in the form of 3mm diameter rods of 20mm length, some of which had a 1mm diameter hole along the cylinder axis, to cope with high cooling rates necessary for some of the experiments. In the case of the welds, the specimens were machined from various positions, as illustrated in Fig. 1, and each specimen was identified accordingly. For the pure alloys, specimens were prepared by swaging, as described below. It was felt that the presence of a

free surface might enhance the nucleation of ferrite, and this was confirmed by cutting one specimen (from each weld) into two 10mm lengths, and plating one of them with a thin layer of nickel; subsequent experiments showed that the specimen not plated with nickel had a transformation start temperature T_h which was $40\text{-}100^{\circ}\text{C}$ higher than the plated specimen, the actual difference increasing slightly with cooling rate but considerably with austenite stabilising alloy content. This is reasonable because nucleation becomes more difficult with alloy content and with fast cooling; hence any enhanced nucleation due to surface effects would be most noticeable in these circumstances. All subsequent experiments were carried out using nickel plated specimens.

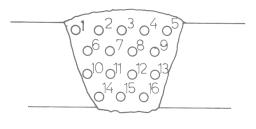


Figure 1 - Identification of dilatometer specimens extracted from submerged arc welds.

The compositions of the alloys used are presented in Table 1. The welds were all prepared by a multipass submerged arc process with about 30 runs in an ISO2560 design joint.

Alloys P1-3 were prepared from high-purity elements as 65g melts in an argon arc furnace on a water-cooled copper mould. The ingots (and some of the specimens machined from welds) were sealed in a quartz tube under a partial pressure of pure argon, and homogenised at 1200°C for 3 days before cold swaging down to 4mm diameter rods. Further cold swaging down to 3mm diameter rod was done in two stages, each preceded by annealing treatments at 600°C for 30 min. The specimens were protected with inert argon during the annealing treatment. The pure alloys were prepared to correspond in C, Si, and Mn concentration to the weld deposits, but their impurity content was meant to be much lower than that of the welds; P1 and P2 have virtually the same C, Si and Mn concentration as W1 and W2 respectively, but unfortunately, P3 has a somewhat lower C concentration than its counterpart W3. This latter discrepancy has been allowed for in the theoretical calculations.

Table 1: Compositions, in wt.%, of welds (W1-3) and pure alloys (P1-3). The concentrations of oxygen and nitrogen are given in parts per million by weight.

No.	C	Si	Mn	Al	S	P	Ο	N
W1	0.10	0.19	1.10	0.015	0.012	0.015	188	115
W2	0.11	0.25	1.57	0.012	0.012	0.018	196	57
W3	0.14	0.22	1.81	0.012	0.013	0.030	138	107
P1	0.10	0.18	1.09	< 0.003	< 0.003	< 0.003	<10	<10
P2	0.11	0.22	1.56	< 0.003	< 0.003	< 0.003	<10	<10
P3	0.09	0.25	1.82	< 0.003	< 0.003	< 0.003	<10	<10

Linear intercept measurements needed to characterise the austenite grain size were carried out using a Quantimet 720 Image Analysing Computer, the very large number of measurements (\approx 200 grains per sample) that this made possible makes the statistical error negligible.

Results and Discussion

Austenite Grain Size

Measurements of the mean lineal intercept are presented in Table 2 below: they refer to the austenite grain structure obtained following reaustenitisation at 1000° C for 10 min. Reaustenitisation involves the nucleation and growth of austenite from a mixture of ferrite and cementite, and after the alloy becomes fully austenitic, the grains may coarsen in order to reduce the total amount of interface present. We note again that this process is not to be compared with the formation of columnar austenite grains from δ -ferrite during the evolution of the primary microstructure of welds.

All of the pure alloys and welds W1 and W3 have approximately the same austenite grain size, but weld W2 for some reason which is not clear shows a significantly higher grain size.

Table 2: Austenite grain size (i.e., mean lineal intercept) obtained after austenitisation at 1000°C for 10 min.

Specimen	Lineal intercept, μm
W1	27
W2	42
W3	17
P1	22
P2	20
P3	21

Transformation Start Temperatures

A typical dilatometric curve is presented in Fig. 2. During cooling from the austenite phase field, the slope of the curve simply represents the expansion coefficient of austenite, which is, found to be constant over the temperature range of interest. By fitting a least squares line to the high temperature part of this curve, the slightest deviation from linearity, caused by the formation of ferrite, can easily be detected; we estimate that this allows the transformation start temperature to be recorded reproducibly to an accuracy of better than 5° C.

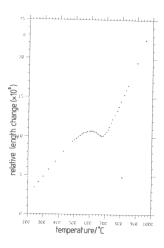


Figure 2 - A typical dilatometric plot of relative length change versus specimen temperature.

During an early stage in the experiments it was found that weld specimens extracted from near the fusion boundary of the parent plate (specimens 1, 5, 6, 9, 10,13, 16 of Fig. 1) had considerably higher T_h temperatures than all of the other specimens. This is because of dilution of the weld by the parent material. Hence, only data from regions of the weld suffering low dilution

are considered further.

The data on transformation start temperatures as a function of cooling rate are presented in Figs. 3-5. Calculated curves, derived from a combination of the additive reaction rule and computed isothermal transformation curves (assuming homogeneous alloys) are also presented in Figs. 3-5.

The results are encouraging; experimental curves for the homogenised welds in all cases correspond well to the calculated curves, although they consistently underestimate T_h by about 10-20°C, the difference decreasing at high cooling rates. The reason for this could be the approximations made in the calculation of the isothermal transformation diagrams or indeed in the use of the additive reaction rule for converting such diagrams to continuous cooling transformation curves. The error caused by an underestimation of T_h by 20°C can be calculated using the microstructure model mentioned earlier (1-4). For a manual metal arc weld of compositions identical to W1, welded at 170A, 21V, 3.6mm/s, interpass temperature 250°C, arc transfer efficiency 0.775, austenite grain size L_{tn} =50µm, the volume fraction of allortiomorphic ferrite changes from 0.38 to 0.37 as T_h changes from 780-760°C. This small change is not surprising since the rate of formation of ferrite is small at T_h .

Comparison of the data from homogenised welds W1 and W2 with alloys P1 and P2 respectively shows that there is excellent agreement between these samples, even though the welds contain considerably higher oxygen levels. This proves that oxygen concentration, upto 200 ppm, does not influence the transformation start temperature of austenite. This is different from published work which we believe is based on an incorrect interpretation of dilatometric curves, from experiments in which the specimens are not protected from effects such as surface nucleation.

The data from homogenised specimen W3 and alloy P3 cannot be compared directly, but when allowance is made for the difference in carbon concentration (Fig. 5), it is clear that both show equally good agreement with calculated data, consistent with the above conclusion.

The effect of alloying element segregation is evident from Figs. 3-5. The transformation start temperatures for the unhomogenised welds are typically $30\text{-}60^{\circ}\text{C}$ higher than those for the homogenised welds, the difference increasing with average alloy content. The amplitude of composition differences in a given alloy must increase with average alloy content, since it is given by the product of the average composition and the partition coefficient during solidification. Thus, the influence of segregation should be most pronounced for more heavily alloyed materials. We note that one important effect of segregation is to increase the amount of allotriomorphic ferrite content of welds, by raising the temperature at which this ferrite first forms. It can be seen from Figs. 3-5 that the procedure for calculating the effect of chemical segregation (7) on T_h works well; the experimental data from the unhomogenised weld agree with the CCT curves calculated for the solute-depleted regions.

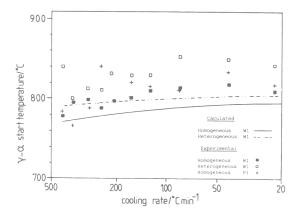


Figure 3 - Calculated and experimental continuous cooling transformation diagrams for specimens W1 (in both the heterogeneous and homogeneous conditions) and P1.

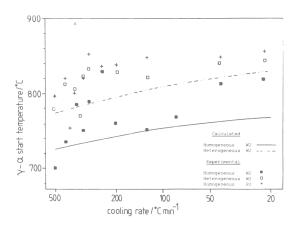


Figure 4 - Calculated and experimental continuous cooling transformation diagrams for specimens W2 (in both the heterogeneous and homogeneous conditions) and P2.

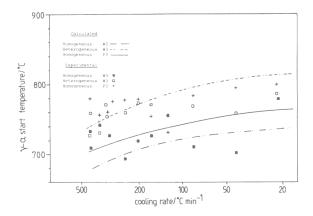


Figure 5 - Calculated and experimental continuous cooling transformation diagrams for specimens W3 (in both the heterogeneous and homogeneous conditions) and P3.

Conclusions

The temperature at which austenite begins to transform diffusionally to allotriomorphic ferrite (T_h) can be measured accurately using high-speed dilatometry, as long as precautions are taken to avoid surface nucleation effects, and effects due to the dilution of the weld by parent material.

A comparison of pure alloys with reheated welds indicates that the presence of oxide inclusions in the latter has no significant effect on T_h .

Continuous cooling curves calculated using thermodynamically predicted isothermal transformation curves combined with an additive reaction rule have been found to be in reasonable agreement with those experimentally obtained from homogenised welds and pure alloys, although the calculations systematically underestimate the transformation start temperatures by some 10-20°C, the discrepancy decreasing with increasing cooling rate.

The presence of alloying element segregation in welds has been shown to raise T_h relative to homogeneous alloys, the difference increasing with average alloy content. The effect can be predicted using a model developed by Gretoft $et\ al.\ (7)$.

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