Kinetics and dilatometric behaviour of non-isothermal ferrite-to-austenite transformation

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1

A model that describes the ferrite-to-austenite transformation during continuous heating in ARMCO iron and three very low carbon low manganese steels with a fully ferrite initial microstructure is presented in this work. This model allows to calculate the volume fraction of austenite and ferrite during transformation as a function of temperature and thus to know the austenite formation kinetics under non-isothermal conditions in full ferritic steels. Moreover, since dilatometric analysis is a technique very often employed to study phase transformations in steels, a second model, that describes the dilatometric behaviour of the steel and calculates the relative change in length which occurs during the ferrite-to-austenite transformation, has been also developed. Both kinetics and dilatometric models have been validated by comparison of theoretical and experimental heating dilatometric curves. Predicted and experimental results are in satisfactory agreement.

1. Introduction

The success of the current research on the modelling of microstructure and properties in steel fabrication depends on the availability of reliable phase transformation theory. A problem of particular importance in this respect is related to the kinetics of austenite formation. Austenitisation is an inevitably occurring during the heat treatment of the vast majority of commercial steels. Despite this consideration, there has been little work carried out on the formation of austenite as compared to the large amount of research on its decomposition. A quantitative theory dealing with the nucleation and growth of austenite from a variety of initial microstructural conditions is vital, especially in the fabrication of steel components by welding.[1]

Early work on austenitisation prior to 1940 was summarised in a paper by Roberts and Mehl,[2] which also reported a study of austenite formation from ferrite/pearlite and ferrite/spheroidite aggregates establishing the nucleation and growth character of the transformation. Posterior works indicated the importance of cementite precipitates in ferrite in aiding nucleation of austenite,[3,4] and considered austenite growth controlled by cementite dissolution.[3,5-7] These investigations give an indication of the complexity of austenite formation in that the austenite is nucleating and growing in a microstructure consisting of two phases which have different degrees of stability.

In the eighties, the development of ferrite/martensite dual phase steels by partial reaustenitisation revived the interest for the heating part of the heat treatment cycle.[8-13] In those works, the authors emphasised the importance of the microstructure immediately before intercritical annealing. Roosz *et al.*[14] determined the influence of

the initial microstructure on the nucleation rate and grain growth of austenite during isothermal treatment of a eutectoid plain carbon steel.

Very little information is available about the austenite formation in steels subjected to continuous heating.[15] Recent work has allowed to model quantitatively the transformation of an ambient temperature steel microstructure into austenite under continuous heating condition.[16] Lately, some investigators have adopted a different approach to the problem using artificial neural network.[17,18]

In this work a model is presented to describe the ferrite-to-austenite transformation during continuous heating in ARMCO iron and three very low carbon low manganese steels with a fully ferrite initial microstructure. This model allows us to calculate the volume fraction of austenite and ferrite during transformation as a function of temperature and thus to know the austenite formation kinetics under non-isothermal conditions in full ferritic steels. Since the ferrite-to-austenite transformation is so rapid in these steels, then reliable measurements of the volume fraction of austenite formed during the partial transformation cannot be obtained experimentally. This renders the validation of the kinetics model through the comparison of the experimental and calculated volume fraction of austenite formed during the progress of the transformation to be impossible. However, dilatometric analysis can be employed as an alternative technique to study phase transformation kinetics in steels. In this sense, the relative change in length which occurs during the ferrite-to-austenite transformation will be calculated as a function of temperature. Both kinetics and dilatometric calculations will be validated by comparing theoretical and experimental heating dilatometric curves.

2. Experimental Procedure

The chemical composition of the studied steels is presented in Table 1. All the steels have a full ferrite microstructure (Fig. 1). Specimens were ground and polished using standardised techniques for metallographic examination. Nital - 2pct etching solution was used to reveal the ferrite microstructure by optical microscopy. The ferrite grain size was measured on micrographs. The average ferrite grain diameter (D) (Table 2) was estimated by counting the number of grains intercepted by one or more straight lines long enough to yield, in total, at least fifty intercepts. The effects of a moderately non-equiaxial structure was eliminated by counting the intersections of lines in four or more orientations covering all the observation fields with an approximately equal weight.[19]

To validate the dilatometric model and also, indirectly, the kinetics model for the ferrite-to-austenite transformation, specimens of 3 mm thick and 12 mm long were heated in a vacuum of 1 Pa at a constant rate of 0.05 K/s in an Adamel Lhomargy DT1000 high-resolution dilatometer. The dimensional variations in the specimen are transmitted via an amorphous silica pushrod. These variations are measured by a linear variable differential transformer (LVDT) in a gas-tight enclosure enabling to test under vacuum or in an inert atmosphere. The heating and cooling devices of this dilatometer were also used to perform all the heat treatments. The DT1000 dilatometer is equipped with a radiation furnace for heating. The energy radiated by two tungsten filament lamps is focused on the dilatometric specimen by means of a bi-elliptical reflector. The temperature is measured with a 0.1 mm diameter Chromel-

Alumel (type K) thermocouple welded to the specimen. Cooling is carried out by blowing a jet of helium gas directly onto the specimen surface. The helium flow rate during cooling is controlled by a proportional servovalve. The high efficiency of heat transmission and the very low thermal inertia of the system ensure that the heating and cooling rates ranging from 0.003 K/s to 200 K/s remain constant.

3. Results and Discussion

3.1. MODELLING OF NON-ISOTHERMAL AUSTENITE FORMATION KINETICS

Formation of austenite from ferrite is well established to be a nucleation and growth process. Speich and Szirmae estimated the maximum ferrite/austenite interface velocity as 0.016 m/s for a 200 μm ferrite grain diameter.[20] This is a very high velocity but still much less than that reported for diffusionless transformations, about 10^3 m/s.[21]

The possible nucleation sites for austenite in pure iron are either in the matrix, at grain boundary faces, at grain boundary edges, or at grain corners.[22] According to Speich and Szirmae's work, matrix nucleation was not detected. All the austenite nucleated at α/α grain boundaries, but grain boundary edges were favoured over grain boundary faces as nucleation sites. Nucleation at corner sites could not be ruled out. However, it was difficult to obtain conclusive evidence for these sites by

examination of two-dimensional sections. This suggests that all nucleation sites should be considered for the modelling.

For grain boundary nucleated phase transformations at sufficiently high nucleation rates, the potential nucleation sites are exhausted early in the transformation, so the reaction is further controlled by growth. This behaviour is called 'site saturation'. Cahn[22] quoted that site saturation occurs when at least one nucleus per grain is formed at a time shorter than D/G, where D is the average ferrite grain diameter and G is the growth rate of austenite into ferrite. Dirnfeld et $al.$[23] calculated that, for an average ferrite grain diameter of 14 μm, one nucleus per grain is formed in a time which is approximately equal to $0.025D/G$ at temperatures between 1033 and 1093 K. Then, consideration of site saturation for the nucleation of austenite into ferrite would be a reasonable approximation in the modelling.

The only available theoretical treatment of boundary migration in the absence of diffusion is that derived from absolute reaction rate theory.[24] Here, the austenite growth is controlled by processes at the interface and the growth rate G is given by:

$$G = \frac{\delta v}{kT}\exp\left(-\frac{\Delta G_{act}}{kT}\right)\Delta g^{\alpha \rightarrow \gamma} = \frac{\delta v}{kT}\exp\left(\frac{\Delta S}{k}\right)\exp\left(-\frac{\Delta H}{kT}\right)\Delta g^{\alpha \rightarrow \gamma}\{T\} \qquad (1)$$

where δ is the boundary thickness, v is the number of attempts to jump the boundary activation barrier per unit time, k is the Boltzman constant, T is the absolute temperature, ΔG_{act} is the free energy for the activated transfer atoms across the ferrite/austenite interface, ΔS is the entropy of activation per atom, ΔH is the enthalpy of activation per atom, and $\Delta g^{\alpha \rightarrow \gamma}$ is the Gibbs free energy difference per

atom between the α and γ phases. The values of ΔH and ν are uncertain but are generally assumed to be equal to the enthalpy of activation for grain boundary diffusion[25] and to kT/h (being h Planck constant), respectively. The value of ΔS is also uncertain and may be negative or positive. If we consider that the maximum ferrite/austenite interface velocity for a 200 μm ferrite grain diameter is 0.016 m/s at 1223 K,[20] $\Delta g^{\alpha \rightarrow \gamma} = 41.87 \times 10^{-24}$ J per atom, $\delta = 5$ Å, and $\Delta H = 276.33 \times 10^{-21}$ J per atom, then $\nu \exp\left(\dfrac{\Delta S}{k}\right)$ is equal to 1.65×10^{17} s^{-1}.

Thus, the growth rate of austenite into ferrite can be calculated as follows:

$$G = \frac{82.3 \times 10^{9}}{kT} \Delta g^{\alpha \rightarrow \gamma} \{T\} \exp\left(\frac{-276.3 \times 10^{-21}}{kT}\right) \tag{2}$$

Figure 2 shows the Gibbs free energy change for the ferrite-to-austenite transformation $\Delta g^{\alpha \rightarrow \gamma}$ for all the studied steels. This energy has been obtained according to the thermodynamic calculations proposed by Aaronson $et\ al.$[26,27] and Kaufman $et\ al.$.[28] In order to account for the effects of alloying elements into calculation, Zener factorization of the free energy into magnetic and non-magnetic components has been performed.[29]

Assuming that site saturation occurs and the reaction is controlled by growth, the kinetics law obtained for the three different activated growth sites can be expressed as follows:[22,23]

$$V_\gamma = 1 - \exp\left[-\left(K_s t + K_e t^2 + K_c t^3\right)\right] \qquad (3)$$

where V_γ represents the formed austenite volume fraction, t is the time and K_s, K_e and K_c are given by,

$$K_s = 2G\frac{S}{V} \qquad K_e = \pi G^2 \frac{L}{V} \qquad K_c = \frac{4}{3}\pi G^3 \frac{C}{V} \qquad (4)$$

where $\frac{S}{V}$, $\frac{L}{V}$ and $\frac{C}{V}$ respectively are the boundary area, the edge length, and the grain corner number, all per unit volume, and G is the growth rate of austenite. Assuming ferrite grains to be tetrakaidecahedra,[22] $\frac{S}{V}$, $\frac{L}{V}$ and $\frac{C}{V}$ can be expressed in terms of the average ferrite grain diameter D by:

$$\frac{S}{V} = \frac{3.35}{D} \qquad \frac{L}{V} = \frac{8.5}{D^2} \qquad \frac{C}{V} = \frac{12}{D^3} \qquad (5)$$

and thus,

$$K_s = \frac{6.7G}{D} \qquad K_e = \frac{26.7G^2}{D^2} \qquad K_c = \frac{50.3G^3}{D^3} \qquad (6)$$

where G is given by equation (2).

9

The difficulties in treating non-isothermal reactions are mainly due to the complex variations of growth rate with temperature, described in equation (2). We can only deal with the problem when the rate of transformation depends exclusively on the state of the assembly and not on the thermal path by which the state is reached.[24] Reactions of this type are called isokinetic. Avrami defined an isokinetic reaction by the condition that the nucleation and growth rates are proportional to each other (i.e. they have the same temperature variation). This leads to the concept of additivity and Scheil's rule.[30]

Since Avrami's condition for an isokinetic reaction is not satisfied in the present case, a general equation to describe the non-isothermal overall ferrite-to-austenite transformation in ferritic steels was derived integrating the equation (3) over the whole temperature range where the transformation takes place.[16] In this sense, we have taken logarithms in equation (3), which then was differentiated,

$$\ln\left(\frac{1}{1-V_\gamma}\right) = K_s t + K_e t^2 + K_c t^3 \tag{7}$$

$$d\left(\ln\frac{1}{1-V_\gamma}\right) = \frac{dV_\gamma}{1-V_\gamma} = \left(K_s + 2K_e t + 3K_c t^2\right)dt \tag{8}$$

If we consider a constant rate, $\dot{T}$, for the heating condition, time can be expressed as follows:

$$dt = \frac{dT}{\dot{T}} \qquad t = \frac{\Delta T}{\dot{T}} \tag{9}$$

substituting into equation (8) and integrating in $\left[0, V_\gamma\right]$ and $\left[T_s, T\right]$ intervals on the

left and on the right sides, respectively, it can be concluded that:

$$\int_0^{V_\gamma} \frac{dV_\gamma}{1-V_\gamma} = \int_{T_s}^{T} \left[K_s + 2K_e \frac{\Delta T}{\dot{T}} + 3K_c \frac{(\Delta T)^2}{\left(\dot{T}\right)^2} \right] \frac{dT}{\dot{T}} \tag{10}$$

thus,

$$-\ln\left(1-V_\gamma\right) = \int_{T_s}^{T} \left[\frac{6.7}{\dot{T}D} G + \frac{53.4}{\left(\dot{T}D\right)^2} G^2\left(T-T_s\right) + \frac{150.8}{\left(\dot{T}D\right)^3} G^3\left(T-T_s\right)^2 \right] dT \tag{11}$$

where T_s is the start temperature of the transformation or temperature at which

$\Delta g^{\alpha \to \gamma} = 0$ (root of the function represented in Fig. 2).

Therefore, the volume fraction of austenite (V_γ) and ferrite (V_α) present in the

microstructure as a function of temperature can be calculated, as follows,

$$V_\gamma = 1 - \exp\left\{ -\int_{T_s}^{T} \left[\frac{6.7}{\dot{T}D} G + \frac{53.4}{\left(\dot{T}D\right)^2} G^2\left(T-T_s\right) + \frac{150.8}{\left(\dot{T}D\right)^3} G^3\left(T-T_s\right)^2 \right] dT \right\} \tag{12}$$

11

$$V_\alpha = 1 - V_\gamma \tag{13}$$

3.2 CALCULATION OF RELATIVE CHANGE IN LENGTH AS A FUNCTION OF TEMPERATURE

Assuming that the sample expands isotropically, the change of the sample length ΔL referred to the initial length L_o at room temperature is related to volume change ΔV and initial volume V_o at room temperature for small changes as follows:

$$\frac{\Delta L}{L_o} = \frac{V - V_O}{3V_O} \tag{14}$$

Therefore, $\dfrac{\Delta L}{L_o}$ can be calculated from the volumes of the unit cells and the volume fractions of the different phases present at every temperature during continuous heating:

$$\frac{\Delta L}{L_o} = \frac{1}{3}\left(\frac{2V_\alpha\, a_\alpha^3 + V_\gamma\, a_\gamma^3 - 2a_{\alpha_o}^3}{2a_{\alpha_o}^3} \right) \tag{15}$$

with

$$a_\alpha = a_{\alpha_o}\left[1 + \beta_\alpha\,(T - 300)\right]$$
$$a_\gamma = a_{\gamma_o}\left[1 + \beta_\gamma\,(T - 300)\right] \tag{16}$$

12

where $V_{\alpha,\gamma}$ are the volume fraction of ferrite and austenite, respectively at any transformation temperature. a_{α_o} is the lattice parameter of ferrite at room temperature, and a_α is the lattice parameter of ferrite at any transformation temperature. The ferrite lattice parameter was taken to be that of pure iron, $a_{\alpha_o} = 2.866$ Å. a_{γ_o} is the lattice parameter of austenite at room temperature as a function of the chemical composition of the austenite, and a_γ is the lattice parameter of austenite at any transformation temperature. $\beta_{\alpha,\gamma}$ are the linear thermal expansion coefficients of ferrite and austenite, respectively, in K^{-1}.

The factor 2 in the numerator of equation (15) are due to the fact that, the unit cell of ferrite contains 2 iron atoms, whereas that of austenite has 4 atoms. The austenite and ferrite volume fractions were calculated at every temperature using equations (12) and (13), respectively. The dependence of the lattice parameter of austenite on alloying elements was taken from Ridley *et al.*[31] and Dyson and Holmes,[32]

$$a_{\gamma_o} = 3.573 + 0.033\text{C} + 0.00095\text{Mn} - 0.0002\text{Ni} + 0.0006\text{Cr} + 0.0031\text{Mo} + 0.0018\text{V} \qquad (17)$$

where the chemical composition is measured in wt-% and a_{γ_o} is in Å.

The values of the linear thermal expansion of ferrite and austenite considered in these calculations were $\beta_\alpha = 1.244 \times 10^{-5}\,\text{K}^{-1}$ and $\beta_\gamma = 2.065 \times 10^{-5}\,\text{K}^{-1}$.[33]

3.3. EXPERIMENTAL VALIDATION OF KINETICS AND DILATOMETRIC CALCULATIONS

The dilatation curves calculated using equation (15) for ARMCO steel and three low carbon low manganese steels (Table 1) with a fully ferrite initial microstructure and heated at a rate of 0.05 K/s are shown in Figs 3 and 4, respectively, in comparison with their corresponding experimental results.

In Fig. 5 the experimental and calculated results of start (T_s) and finish (T_f) temperatures of ferrite-to-austenite transformation are compared. T_s is considered to be the temperature at which the relative change in length of the steel deviates from a linear relation with temperature during heating due to the formation of austenite; T_f has been defined as the temperature at which the sample exhibits again a linear thermal expansion relation once the ferrite-to-austenite transformation is completed. Points lying on the line of unit slope show a perfect agreement between experimental and calculated values.

The calculated curves shown in Figs 3 and 4 suggest that the ferrite-to-austenite transformation takes place almost instantaneously (1 K). In contrast, the experiments reveal that this transformation needs between 10 and 20 K to reach completion at a heating rate of 0.05 K/s.

Additionally, Figs 3, 4 and 5 show that experimental T_s and T_f temperatures are higher than those predicted for all the studied steels. Any difference between these represents some kinetic hindrance to transformation. Fig. 5 shows that the ARMCO steel transforms to austenite at temperatures which are similar to the predicted temperatures. The addition of manganese clearly leads to much larger deviations

from calculated results. That may be explained by the fact that the presence of a substitutional solute retards the transformation to austenite because it is necessary for the solute to diffuse during transformation.[17]

In general, the calculated relative change in length was consistent with the measured value at every temperature. The fact that both the modelled and the experimental dilatometric curves run parallel is irrelevant as long as the adequate hermal expansion coefficients are calculated adequately.[16] The linear expansion coefficients of ferrite and austenite from Takahashi[33] are in a good agreement with those measured values.

4. Conclusions

1. A mathematical model applying the Avrami equation has been successfully developed to reproduce the kinetics of the ferrite-to-austenite transformation in ferritic steels during continuous heating. Nucleation of austenite occurs at the α/α grain boundaries. All possible nucleation sites at the grain boundaries have been taken in consideration in the modelling assuming that no nucluation barrier exists. Since ferrite/austenite boundary migrates in the absence of diffusion, the growth of austenite has been considered to be controlled by processes at the interface.

2. The relative change in length which occurs during the ferrite-to-austenite transformation has been calculated as a function of temperature. Experimental values of relative changes in length and linear expansion coefficients measured by high resolution dilatometry are accurately reproduced by calculations.

3. Experimental validation of the kinetics model of the ferrite-to-austenite transformation has been carried out by comparison between experimental and theoretical heating dilatometric curves. Results show that experimental start and finish temperatures of the transformation are higher than those predicted for all the studied steels. Furthermore, the addition of manganese clearly leads to much larger deviations from calculated results since the presence of a substitutional solute retards the transformation to austenite.

5. Acknowledgements

6. References

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Table 1   **Chemical Composition (Mass %)**

Steels	C	Mn	Si	N	Al	P	Cr	Ni
ARMCO	0.002	0.05	-	0.004	-	0.003	-	-
C-0.25Mn	0.010	0.25	0.028	0.003	0.057	0.016	0.014	0.022
C-0.37Mn	0.010	0.37	0.028	0.002	0.069	0.016	0.016	0.022
C-0.50Mn	0.010	0.50	0.028	0.004	0.046	0.015	0.012	0.020

Table 2   **Average Ferrite Grain Diameter**

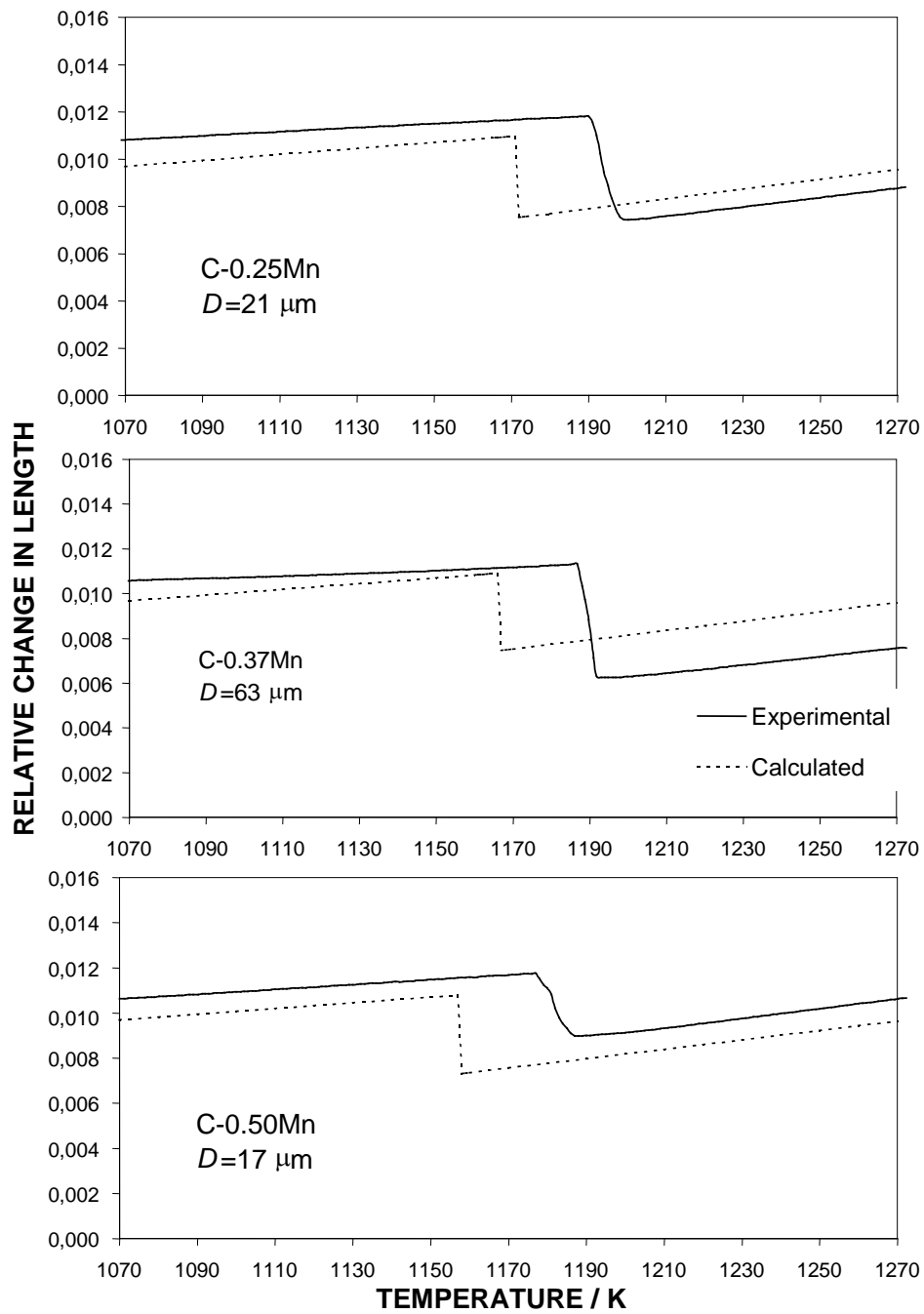| Steels	D / μm
ARMCO	158±28
C-0.25Mn	21±3
C-0.37Mn	63±11
C-0.50Mn	17±1
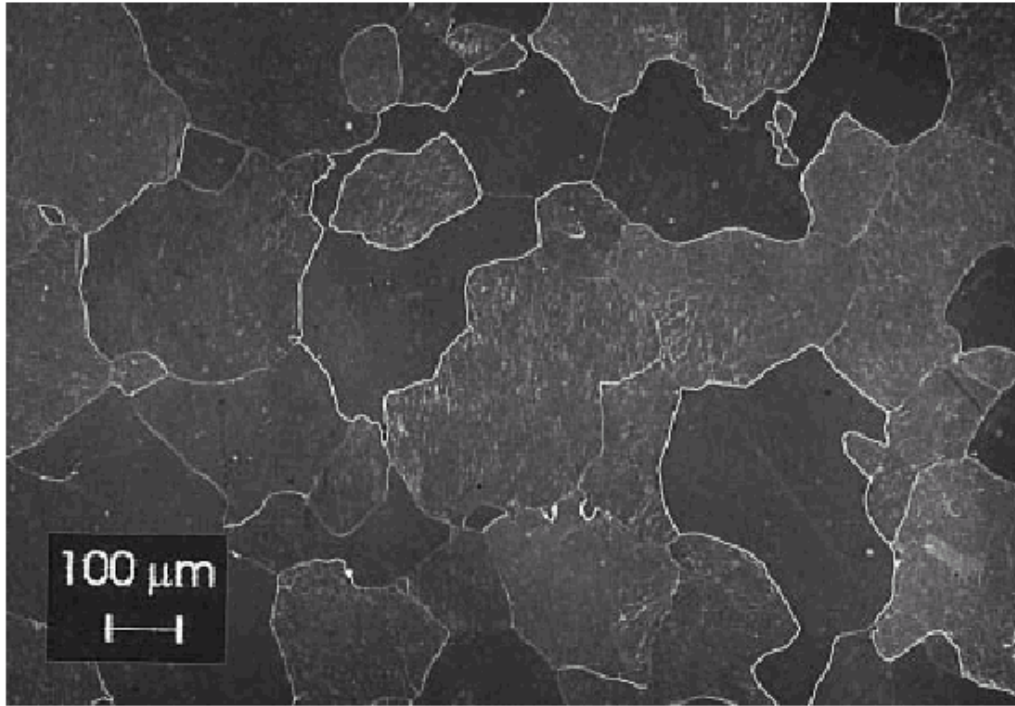
**1 Optical micrograph of a full ferrite microstructure. ARMCO steel**
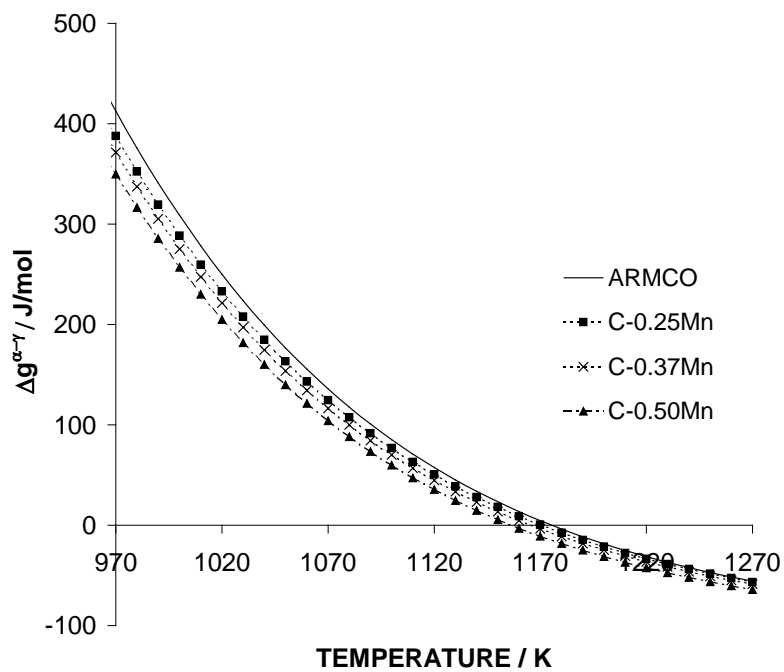
2 Gibbs free energy change for $\alpha\rightarrow\gamma$ transformation
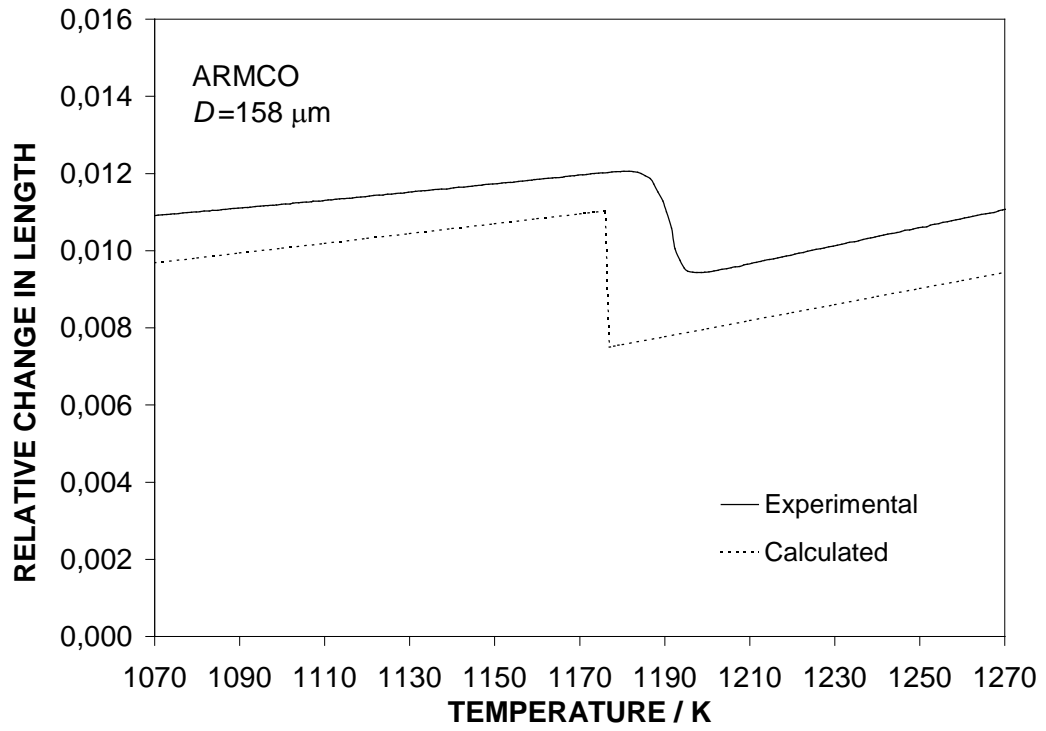
3 Calculated dilatation curve of ARMCO steel compared with the experimental curve obtained at a heating rate of 0.05 K/s

4 Calculated dilatation curve of three low carbon low manganese steels compared with their corresponding experimental curves obtained at a heating rate of 0.05 K/s
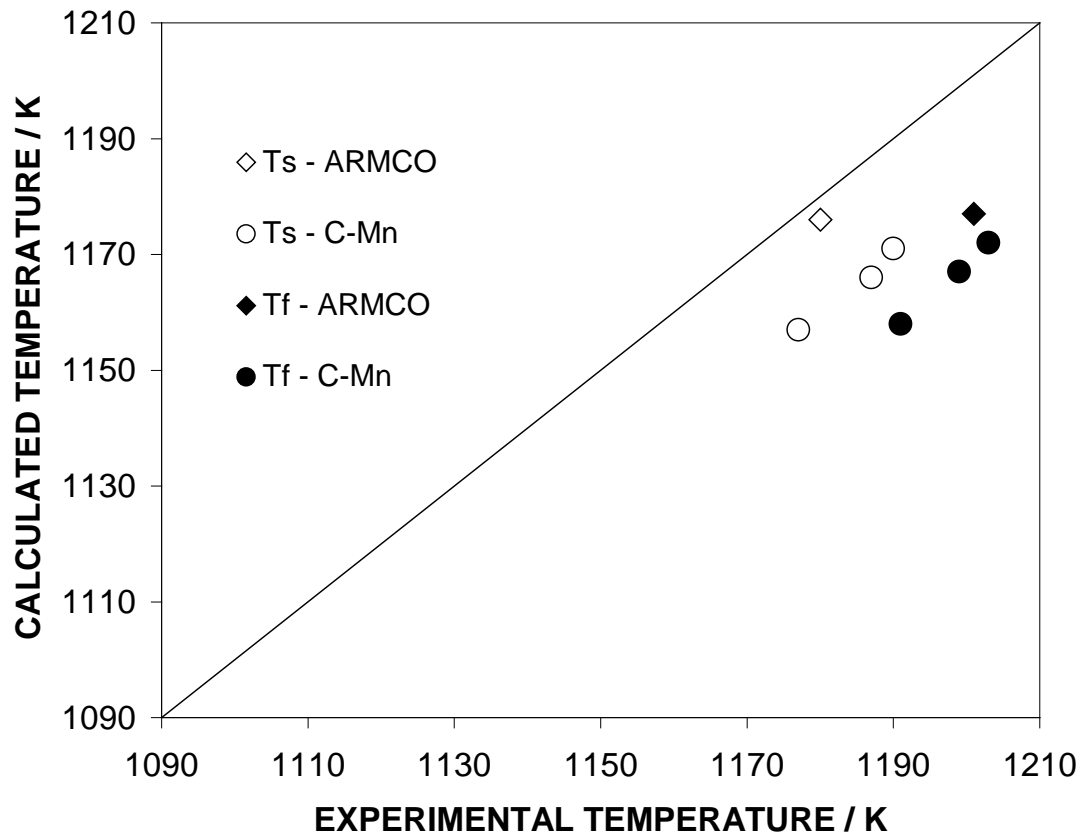
5 Comparison of experimental and calculated start (T_s) and finish (T_f) temperatures of ferrite-to-austenite transformation