In-situ study of austenite formation by dilatometry in a low carbon microalloyed Steel

D. San Martín[1,2], P. E. J. Rivera-Díaz-del-Castillo[1], C. García-de-Andrés[2].

[1]Fundamentals of Advanced Materials Group, Faculty of Aerospace Engineering, Delft University of Technology, Kluyverweg 1, 2629 HS Delft, The Netherlands. Website: www.fam.lr.tudelft.nl/ .

[2]MATERALIA Research Group, Department of Physical Metallurgy, Centro Nacional de Investigaciones Metalurgicas (CENIM-CSIC), Av. Gregorio del Amo 8, 28040, Madrid, Spain. Website: http://www.cenim.csic.es/materalia/ . Email: dsm@cenim.csic.es

Abstract: In-situ studies of reaustenitization in a low carbon microalloyed steel have been carried out by dilatometry. A model is proposed for describing the temperature evolution of the austenite volume fraction and its carbon concentration during continuous heating for various heating rates from dilatometric plots. The model results match very well metallographic measurements and may be applied to the reaustenitization of low carbon steels.

Keywords: High strength Low Alloy (HSLA) steel, Dilatometry, Phase transformations, Austenite.

The formation of austenite from ferrite/pearlite aggregates is a process of great technological importance in multiphase steels possessing a compromise between properties such as strength and ductility [1]. Usually referred to as reaustenitization, in this heating process the initial microstructure dissolves in favor of austenite. Through variation of the heating rate, it becomes possible to control the volume fraction, composition and grain sizes of austenite and ferrite. These factors critically influence further transformations on cooling such as the formation of martensite from austenite on quenching [2] or the formation of bainite at lower temperatures [3]. A number of physically based models can be found in the literature to predict the time and temperature evolution of the austenite volume fraction [4-6]. There is, however, a shortfall in reliable techniques for obtaining consistent experimental data to test those models. The present contribution provides a semi-empirical technique to quantify phase kinetics during reaustenitization.

Methods and techniques to determine the volume fraction of phases avoiding the time-consuming metallographic examination of samples constitute important tools for metallurgists. On the other hand, quantitative metallography studies on austenite formation require an interrupted heat treatment by quenching. This process may be accompanied by inaccuracies as other phases such as bainite and martensite may form, obscuring the metallographic observations at room temperature.

Some of the most usual experimental techniques for the in-situ study of phase transformations in steels are differential scanning calorimetry [7] and magnetization measurements [8]; however, dilatometry is probably the most widely used technique [9-16]. There exist semi-empirical models to quantify the progress of ferrite and/or pearlite on cooling [13,14,17]. Nevertheless, none of these approaches has tried to estimate the volume fraction of austenite from dilatometric data on continuous heating in hypoeutectoid steels. A semi-empirical method has been developed in this investigation to estimate the temperature evolution of the austenite volume fraction. This has been applied to a low carbon steel during a continuous heating at a constant rate. Moreover, the temperature evolution of the carbon concentration in austenite has also been estimated by accounting for mass conservation during the transformation. These results have been compared to metallographic measurements finding a very good agreement.

2

The steel investigated in this work (0.11C-1.5Mn-0.3Si-0.02P-0.03Nb-0.01Cu-0.04Al-0.005N, wt-%) has a pearlitic-ferritic initial microstructure. The initial pearlite volume fraction (0.13) present in the microstructure has been determined by the point counting method. The formation of austenite during continuous heating has been analyzed by an Adamel Lhomargy DT1000 high resolution dilatometer, with sample dimensions of 2 mm in diameter and 12 mm length.

In order to track the evolution of the volume fraction of austenite during continuous heating the critical transformation temperatures Ac_1 and Ac_3 have been determined from dilatometric plots. These temperatures establish the start and finish of austenite formation. Four different heating rates have been studied in this work (0.05, 0.5, 5, 10 °C/s). Figure 1 shows a characteristic dilatometric curve, after continuous heating at 0.05 °C/s, where the critical transformation temperatures are shown. After these temperatures were established, several quench-out temperatures were selected between Ac_1 and Ac_3 to study the progress of austenite formation during heating. The volume fraction of martensite present in the microstructures after quenching is assumed to be equal to that of austenite at high temperature prior to quenching. Martensite was revealed by LePera etching solution [18] and the volume fraction determined by the point-counting method. Bainite formation during quenching was only observed after interrupted heating by quenching at temperatures very close to Ac_3. In these cases, Nital-2% gave better contrast to differentiate between proeutectoid ferrite (present in the initial microstructure) and bainite/martensite (transformed on quenching from austenite). The temperature evolution of the austenite volume fraction was determined metallographically for every heating rate and is shown in Figure 2 as open markers. Table 1 gives the value of temperatures Ac_1 and Ac_3 for each heating rate. These results were obtained after averaging the values of eight different dilatometric curves per heating rate.

During austenite formation, the local changes in the crystal structure result in a macroscopic volume contraction of the sample. These changes can be detected and quantified by dilatometry. The volume contraction has two main contributions: 1) the difference in specific volume between the phases involved in the transformation (austenite, ferrite and cementite) and 2) the variation of the austenite specific volume due

3

to the carbon enrichment or depletion. Very long heating times just above Ae_1 are generally necessary to have equilibrium partitioning of substitutional alloying elements like Mn [19, 20]. However, incomplete or partial partitioning may also occur above this temperature depending on the heating rate. In this work it is considered that the transformation takes place under paraequilibrium conditions [21], the diffusion of carbon is assumed to be the rate controlling factor for the formation of austenite and it is considered that no partitioning of other alloying elements takes place during the transformation. Moreover, it is assumed that austenite carbon content during pearlite to austenite transformation remains, in average, constant and equal to the eutectoid composition of the steel, although it is unlikely that this composition will be equal to the eutectoid one when the pearlite has formed at temperatures approaching Ae_1 [22].

To undertake the conversion of dilatometric data into volume fraction transformed, the austenitization process has to be split in two separate but consecutive steps, i) pearlite to austenite and ii) ferrite (α) to austenite (γ). The microstructure of this steel contains cementite precipitates at ferrite grain boundaries. These precipitates represent a very small fraction of the total volume fraction of cementite (mainly present at pearlite colonies). It has been observed that only at low heating rates (0.05 ºC/s) some nuclei of austenite form at these particles. The contribution to the volume fraction of austenite formed during the pearlite to austenite transformation in the first step of the transformation is only around 2% [23], so the overlapping between both transformations is small. The formation of austenite at ferrite grain boundaries during the first step of the transformation has not been taken into account. The transition temperature between both transformations (Ac_θ) can be derived from the dilatometric curve as the temperature at which the first contraction due to pearlite dissolution finishes (see Figure 1). At this temperature it is considered that the amount of austenite transformed equals the amount of pearlite in the starting microstructure. Previous experimental investigations in low carbon steels support this assumption [24, 25]. Ac_θ has been determined for every heating rate and is given in Table 1. Again, the results of eight different dilatometric curves per heating rate have been used to estimate this temperature.

4

For the higher heating rates studied in this work (>5 °C/s), very small amount of cementite has been observed to remain undissolved after the dissolution of pearlite nodules and, when present, it does not seem to affect the dilatometry plots strongly so its effect has been considered to be weak. Its influence has not been considered in this model.

As described before, during the first step of the transformation the nucleation and growth of austenite from pearlite takes place. It is considered that the volume fraction of ferrite remains constant ($f_{\alpha,0} = 0.87$). The average atomic volume of the sample (V) and the volume fraction of austenite (f_γ) can be expressed as,

$$V = f_{\alpha,0}V_\alpha + f_\gamma V_\gamma + f_p V_p, \tag{1}$$

$$f_\gamma = 1 - f_{\alpha,0} - f_P. \tag{2}$$

Where V_α, V_γ and V_P represent the atomic volume of ferrite, austenite and pearlite, respectively, and f_P the volume fraction of pearlite. The atomic volume of pearlite can be expressed as $V_p = (1-\rho)V_\alpha + \rho V_\theta$; with V_θ and $\rho = 1/9$ the atomic volume of cementite (θ) and the fraction of cementite in pearlite, respectively. Combining equations (1)-(2), the following expression is derived,

$$f_\gamma = \frac{V - V_\alpha + f_{p,0}\left(V_\alpha - V_p\right)}{\left(V_\gamma - V_p\right)}. \tag{3}$$

Considering that the carbon content of the steel is 0.11 wt-%, the carbon concentration in austenite χ_C^γ during this transformation can be estimated from the mass conservation as,

$$\chi_C^\gamma = \frac{0.11 - f_\alpha \chi_C^\alpha}{f_\gamma}. \tag{4}$$

From equation (4), a value of $\chi_C^\gamma = 0.68$ wt-% at the end of pearlite dissolution results from $f_\gamma = 0.13$, $f_\alpha = 0.87$ and by assuming a constant carbon content in ferrite of $\chi_C^\alpha = 0.025$ wt-%. Once all pearlite has dissolved, high carbon austenite grains (of eutectoid composition) start consuming the low carbon ferrite remaining in the microstructure. For this second step of the transformation, equations (1)-(3) can now be written as,

$$V = f_\alpha V_\alpha + f_\gamma V_\gamma . \tag{5}$$

$$f_\gamma = 1 - f_\alpha . \tag{6}$$

$$f_\gamma = \frac{V - V_\alpha}{V_\gamma - V_\alpha} . \tag{7}$$

In equations (3) and (7) the atomic volume of each phase is a function of the lattice parameters of ferrite (a_α), austenite (a_γ) and cementite (a_θ, b_θ, c_θ), which can be written as $V_\alpha = a_\alpha^3 / 2$, $V_\gamma = a_\gamma^3 / 4$ and $V_\theta = a_\theta b_\theta c_\theta / 12$. The variation of the lattice parameters with temperature and composition was determined by Onink et al. [26].

The atomic volume of the sample can be written as a function of the relative change in length,

$$V = kV_0 \left(3 \frac{\Delta l}{l_0} + 1 \right), \tag{8}$$

where V_0 is the initial atomic volume of the sample, Δl and l_0 are the variation in length along the longitudinal axis and the initial height of the cylindrical sample, respectively, and k is a scaling factor. The value of this factor is ideally 1, but a number of reasons that make it to depart from 1: inaccuracies in the measuring system, non-isotropic contraction/expansion in the sample due to the presence of texture and transformation induced plasticity effects. These effects are not considered in the model. The value of k

has to satisfy that the value of f_γ, calculated from equations (3) and (7), is $f_\gamma = 0$, $f_\gamma = f_p^0 = 0.13$ and $f_\gamma = f_p^0 + f_\alpha^0 = 1$, at Ac_1, Ac_θ and Ac_3, respectively. Due to the unknown temperature dependence of *k*, it is usually assumed to increase linearly between the start and the end of the transformation [12,13]. In the present work values of 0.99<k<1.01 were considered.

Equations (3), (4) and (7) were numerically solved for f_γ and χ_C^γ in temperature steps of ~0.75 °C. Figure 2 shows the evolution of the average austenite volume fraction estimated by this method. Eight dilatometric curves have been analyzed for every heating rate. There is a very good correlation between the estimations of the dilatometric model and the experimental values obtained from metallography (open markers). Figure 3 shows the temperature evolution of the carbon content in austenite for the different heating rates. The calculations shown in this figure have been undertaken by using equation (4); the value of the open markers have been estimated from the metallographic austenite volume fraction measurements given in Figure 2, and the solid lines have been estimated by averaging the results obtained for eight different dilatometric curves.

Figures 2 and 3 show very good agreement between the results of this method and the metallographic measurements, suggesting that the model presented in this work can be successfully implemented to convert dilatometric data obtained from a continuous heat treatment into austenite volume fractions in hypoeutectoid steels with an initial microstructure formed by pearlite and ferrite. This approach assumes that the pearlite to austenite and the ferrite to austenite transformations occur in successive steps, that the transformation takes place under paraequilibrium (only carbon partitions), that pearlite has the eutectoid carbon composition and that cementite dissolution is complete when pearlite has fully transformed into austenite; moreover, cementite particles present at ferrite grain boundaries are neglected. In applying this method, the detection of the first contraction due to pearlite dissolution is essential (Figure 1) for estimating the location of Ac_θ temperature. In a previous study [23], it has been reported that such detection stems from the interlamellar spacing of pearlite: a finer microstructure results in a faster dissolution process and the enhancement of this contraction with respect to the overall transformation, compared to coarser microstructures.

7

References

[1] M. Militzer, Science 298 (2002) 975-976.

[2] E. Jiménez-Melero, N. H. van Dijk, L. Zhao, J. Sietsma, S.E. Offerman, J.P. Wright and S. van der Zwaag, Scripta Mater. 56 (2007) 421-424.

[3] H. K. D. H. Bhadeshia, Bainite in steels, The institute of Materials, London, UK, 1992.

[4] F. G. Caballero, C. Capdevila, D. San Martín, C. García-de Andrés, Rev. Metal. Madrid 40 (2004) 214-218.

[5] P. E. J. Rivera Diaz del Castillo, S. van der Zwaag, Proceedings of the International Conference on Solid-Solid Phase Transformations In Inorganic Materials (PTM 2005), Arizona, Vol. 2, 801-810.

[6] A. Jacot and M. Rappaz, Acta Mater. 47 (1999) 1645-1651.

[7] M. Guner, E. Guler, E. Yasar, H. Aktas, Physica B 395 (2007) 16-19.

[8] D. San Martin, N. H. van Dijk, E. Bruck, S. van der Zwaag, Mater Sci. Eng. A (2007), doi:10.1016/j.msea.2006.11.177.

[9] F. G. Caballero, C. Capdevila, D. San Martín, C. García de Andrés, Rev. Metal. Madrid 40 (2004)214-218.

[10] T. de Cock, C. Capdevila, F. G. Caballero, C. García de Andrés, Scripta Mater. 54 (2006) 949-954.

[11] F. G. Caballero, L. F. Álvarez, C. Capdevila, C. García de Andrés, Scripta Mater. 49 (2003) 315-320.

[12] C. García de Andrés, F. G. Caballero, C. Capdevila, L. F. Álvarez, Mater. Charact. 48 (2002) 101-111.

[13] M. Gómez, S.F. Medina, G. Caruana, ISIJ International 43 (2003) 1228-1237.

[14] T. A. Kop, J. Sietsma, S. van der Zwaag, J. Mater. Sci. 36 (2001) 519-526.

[15] F. G. Caballero, C. Capdevila, C. García de Andrés, Mater. Sci. Technol. 17 (2001) 1114-1118.

[16] F.G. Caballero, C. Capdevila, C. García de Andrés, ISIJ International, 41 (2001) 1093-1102.

[17] S. Choi, Mater. Sci. Eng. A363 (2003) 72-80

[18] F. S. LePera: J. Met. 32 (1980) 38-39

[19] C. I. Garcia and A. J. DeArdo, Metall. Trans. A 12A (1981) 521-530.

[20] G. R. Speich, V. A. Demarest, and R. L. Millar, Metall Trans. A 12A (1981) 1419-1428.

[21] H. K. D. H. Bhadeshia, Prog. Mater. Sci. 29 (1985) 321.

[22] P. R. Howell, Mater Charact. 40 (1998) 227-260

[23] D. San Martín, T. de Cock, A. García-Junceda, F. G. Caballero, C. Capdevila, C. García de Andrés, Mater. Sci. Tech., Accepted 2007.

[24] C. García de Andrés, F. G. Caballero, C. Capdevila, Scripta Mater. 38 (1998) 1835-1842.

[25] F. G. Caballero, C. Capdevila, C. García de Andrés, J. Mater. Sci. 37 (2002) 3533-3540.

[26] M. Onink, C.M. Brakman, F. D. Tichelaar, E. J. Mittemeijer, S. van der Zwaag, Scripta Metall. Mater. 29 (1993) 1011-1016.
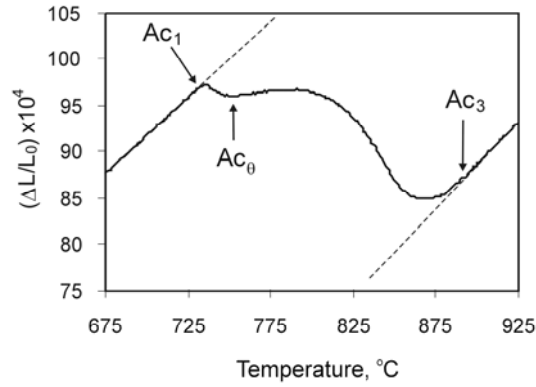
Figures

Figure 1. Characteristic dilatometric curve obtained after heating at 0.05 °C/s.
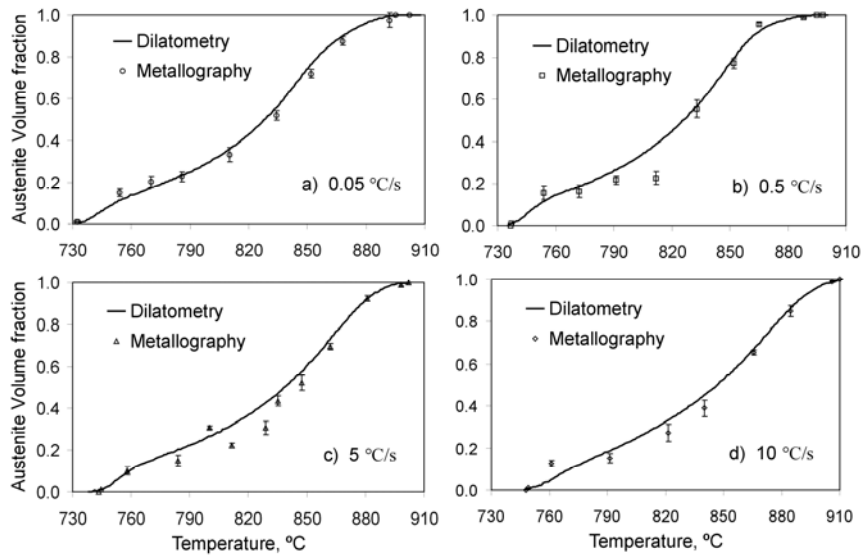

Figure 2. Temperature evolution of the volume fraction of austenite for four different heating rates. Open markers have been determined by metallographic measurements and the solid lines have been estimated from dilatometric data.
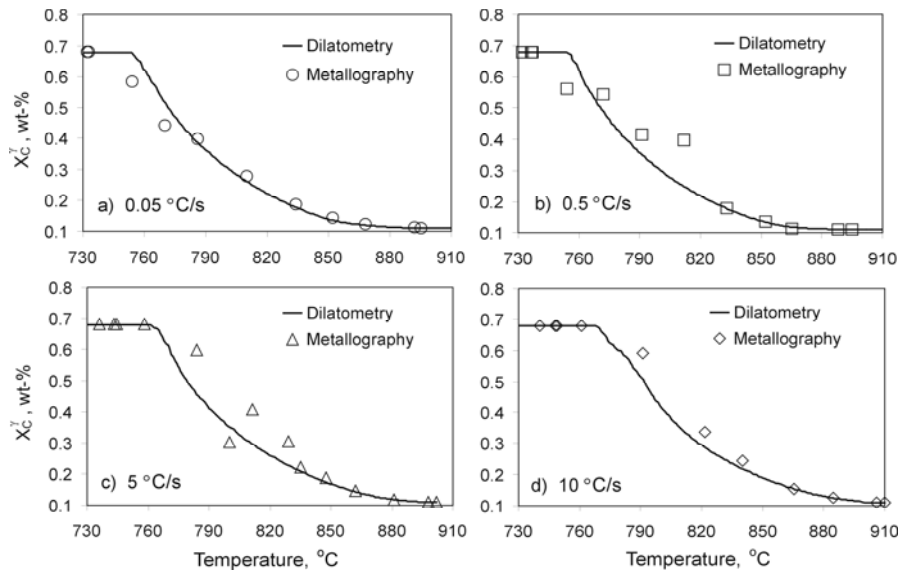
Figure 3. Temperature evolution of the carbon concentration in austenite for the four different heating rates. Open markers have been estimated from the metallographic austenite volume fraction measurements shown in Figure 2 by using equation (4). Solid lines have been estimated from dilatometric curves by using the same equation.

Tables:

Table 1. Experimental start (Ac_1) and end (Ac_3) temperatures of austenite formation for each heating rate. The experimental estimation of the pearlite dissolution finishing temperature (Ac_θ) is also given.

Heating rate, ºC/s	Ac_1, ºC	Ac_θ, ºC	Ac_3, ºC
0.05	732±1	756±4	893±4
0.5	736±2	756±2	889±6
5	742±2	763±4	897±5
10	752±4	776±5	905±5