

INCUBATION TIME OF ISOTHERMALLY TRANSFORMED ALLOTRIOMORPHIC FERRITE IN MEDIUM CARBON STEELS

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Introduction

Medium carbon microalloyed steels have become of great interest because of the possibility to form mainly acicular ferrite microstructures after the forging process of these steels. An important improvement in toughness has been reached in these steels as comparison with those with a conventional ferrite-pearlite microstructure [1-5]. These steels are developed for the automotive, offshore oil and gas industries, and are designed to be in service in corrosive environment.

In medium carbon microalloyed steels, acicular ferrite is always formed after the growth of allotriomorphic ferrite and pearlite [4]. As a consequence, acicular ferrite transformation is expected to be influenced by the previous allotriomorphic ferrite formation. The role of the allotriomorphic ferrite to promote the formation of acicular ferrite to the detriment of bainite has been reported in previous works [1,6-8]. Thus, the amount of acicular ferrite increases, as allotriomorphic ferrite is present along the austenite grain boundaries of medium carbon microalloyed steels. Therefore, a deep understanding of the decomposition of austenite in allotriomorphic ferrite is needed in order to control the total amount of acicular ferrite. In this sense, a theoretical prediction of the incubation time for allotriomorphic ferrite transformation can be useful for the development of medium carbon microalloyed steels.

A great number of attempts, from purely empirical to semiempirical models, have been made to predict the start of ferrite transformation [9-10]. However, models recently developed are becoming less empirical since they rely on thermodynamic and phase transformation theories [11]. In principle, most of these models are able to predict the incubation time of ferrite allotriomorphs for low carbon low alloy steels. But, the level of agreement between predicted and calculated incubation times for allotriomorphic ferrite is less satisfactory for medium carbon steels. The purpose of this study is to predict the incubation time for allotriomorphic ferrite formation in medium carbon microalloyed steels. The influence of alloying elements on carbon diffusion of in austenite has been considered.

Experimental Procedure

The chemical composition of three different medium carbon steels is listed in Table 1. A conventional carbon manganese steel (CMn), and two different microalloyed steels (VTi1 and VTi2) with very different Ti content have been considered in this study. The isothermal decomposition of austenite has been analysed by means of an Adamel Lhomargy DT1000 high-resolution dilatometer described elsewhere [12]. The heating device consists of a very low thermal inertia radiation furnace. The heat radiated by two tungsten filament lamps is focussed on the specimen (cylindrical test pieces of 2-mm in diameter and 12-mm in length) by means of a bi-elliptical reflector. The temperature is measured with a 0.1 mm in diameter, Chromel–Alumel (Type K) thermocouple spot welded to the specimen. Blowing a jet of helium gas directly onto the specimen surface

induces cooling. The helium flow rate during cooling is controlled by a proportional servovalve. These devices ensure an excellent efficiency in controlling the temperature and holding time of isothermal treatments and fast cooling in quenching processes.

TABLE 1
Composition of steels (wt. %)

Steels	C	Mn	N	Si	Cu	Cr	Ni	Ti	V	Mo
CMn	0.31	1.22	-	0.253	-	0.138	0.098	-	0.004	0.03
VTi1	0.37	1.45	0.0162	0.56	0.14	0.04	-	0.015	0.11	0.025
VTi2	0.32	1.39	0.0148	0.33	0.129	0.13	0.14	0.039	0.129	0.03

As it is well known, prior austenite grain size (PAGS) exerts an important influence on the decomposition of austenite [13]. Austenitisation conditions were selected to achieve approximately the same PAGS in all the steels (Table 2). After austenitisation, specimens were isothermally transformed at temperatures in the range of 973 to 873 K at different times and subsequently were gas-quenched under helium gas flow at a cooling rate of 200 K/s.

TABLE 2
Austenitisation Conditions

Steels	Temperature, (K)	Time, (s)	PAGS, (nm)
CMn	1473	120	70
VTi1	1523	60	76
VTi2	1523	180	72

Specimens were ground and polished using standardised metallographic techniques. Nital-2pct etching solution was used to reveal the ferrite microstructure by optical microscopy. The PAGS was calculated on recorded micrographs by using a linear intercept technique, involving at least 50 intercepts, permitting the count of the number of grains intercepted by the grid line. The effects of a moderately non-equiaxial structure may be eliminated by counting the intersections of lines in four or more orientations covering all the observation fields with an approximately equal weight [14].

Results and Discussion

According to the classical nucleation theory, the nucleation rate for the allotriomorphs, I , is expressed as follows [15]:

$$I = I_0 \exp\left(-\frac{t}{\tau}\right) \exp\left(-\frac{U^*}{k_B t}\right) \quad (1)$$

where I_0 is the steady state nucleation rate, t is the isothermal reaction time, and τ is the incubation time. Lange *et al.* [16] proposed a model to calculate I based on a traditional disk-shaped 'pillbox' nucleus. In that case, I is estimated as follows,

$$I = I_0 \exp\left(-\frac{12k_B T a^4 s_{ag}}{D_C^g n_a^2 D G_v^2 t}\right) \quad (2)$$

where k_B is Boltzmann constant; D_C^g is the diffusivity of carbon in austenite; n_a is the volume of an atom of iron in ferrite; a is the average of the lattice parameters of both phases, ferrite and austenite for a given

temperature and steel composition; \bar{x} is the average carbon content of the steel in mole fraction; DG_v is the volume free energy change associated to the formation of the nucleus; s_{ag} is the interfacial energy of a disorder ferrite; and T is the isothermal temperature. From a comparison between equations (1) and (2), an expression for the incubation time can be obtained straightforward:

$$t = \frac{12k_B T a^4 s_{ag}}{D_C^g \bar{x} n_a^2 DG_v^2} \quad (3)$$

The theoretical determination of D_C^g , performed by Siller and McLellan [17] and reviewed by Bhadeshia [18], considers both the kinetic and equilibrium thermodynamic behaviour of carbon in austenite. Calculation of D_C^g takes also into account two important factors: the concentration dependence of the activity of carbon in austenite [19] and the repulsive interactions between nearest neighbouring carbon atoms located in octahedral interstitial sites [20]. Thus, the diffusivity D_C^g is calculated by two factors, a concentration dependent factor and a concentration independent factor,

$$D_C^g = \chi(q) \frac{k_B T}{h} \frac{\exp\left(-\frac{DG^*}{k_B T}\right)}{3g_m} \quad (4)$$

where $\chi(q)$ is the carbon concentration dependent factor obtained according to Bhadeshia's calculations [18] and takes values listed in Table 3; DG^* is the activation free energy for diffusion which is independent of composition and temperature; g_m is an activity coefficient assumed constant; l is the distance between the {002} austenite planes and h is the Planck's constant. Bhadeshia [18] found that $DG^* / k_B = 21230$ K and $\ln\{g_m / l^2\} = 31.84$. Likewise, Babu and Bhadeshia [21] completed this model analysing the effect of different substitutional alloy elements on the diffusivity of carbon in austenite. The values of D_C^g for temperatures ranging from 973 K to 873 K are listed in Table 4.

TABLE 3
Calculated Values of $\chi(q)$ Factor at Different Temperatures for the Three Steels

T, (K)	CMn	VTi1	VTi2
973	0.007	0.009	0.011
948	0.012	0.013	0.012
923	0.014	0.015	0.014
898	0.016	0.017	0.016
873	0.019	0.023	0.011

The volume free energy change, DG_v , in equation (3) has been calculated with the help of a commercial software namely MTDATA [22], which contains a large and rigorously evaluated thermodynamic database. DG_v calculations take into account the effect of all the alloying elements in the decomposition of austenite. The values obtained for the studied steels are listed in Table 4.

In order to determine a in equation (3), it is needed to calculate previously the lattice parameters of ferrite and austenite. The ferrite lattice parameter has been estimated using the relationships given by Leslie [23] and Bhadeshia [24], which take into consideration the equilibrium dissolved carbon in ferrite as well as the different substitutional alloying elements. On the other hand, the austenite lattice parameter has been calculated as reported by Ridley *et al.* [25] and Dyson and Holmes [26] considering the dependence of the lattice parameter of austenite on alloying elements. The values of a at temperatures ranging from 973 K to 873 K are also listed in Table 4.

TABLE 4

Calculated Values of DG_v , D_C^g and a Parameters at Different Temperatures for the Three Steels

T (K)	CMn			VTi1			VTi2		
	$D_C^g \cdot 10^{-14}$ ($m^2 s^{-1}$)	$DG_v \cdot 10^7$ ($J m^{-3}$)	a (pm)	$D_C^g \cdot 10^{-14}$ ($m^2 s^{-1}$)	$DG_v \cdot 10^7$ ($J m^{-3}$)	a (pm)	$D_C^g \cdot 10^{-14}$ ($m^2 s^{-1}$)	$DG_v \cdot 10^7$ ($J m^{-3}$)	a (pm)
973	24.0	-1.62	330.5	30.9	-0.96	330.6	36.4	-1.11	331.7
948	22.5	-2.69	330.4	24.3	-2.00	330.5	22.7	-2.07	331.5
923	13.7	-3.83	330.2	14.8	-3.23	330.3	13.8	-3.21	331.4
898	8.1	-5.30	330.0	8.7	-4.48	330.2	8.2	-4.53	331.2
873	4.6	-6.03	329.9	5.8	-5.94	330.0	2.7	-6.28	331.1

Likewise, a s_{ag} value of $0.705 J m^{-2}$ [16,27] and an atomic radius of iron atom into ferrite lattice of 128 pm [28] ($n_a = 8.785 \cdot 10^{-30} m^3$) have been considered in equation (3) for t calculations.

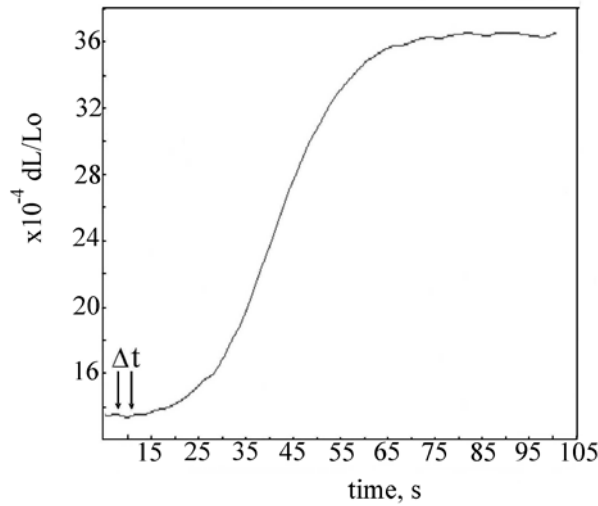


Figure 1. Dilatometric curve (relative change in length vs time) obtained during isothermal decomposition of austenite at 873 K during 100 s for VTi1 steel.

Figure 2. Optical micrograph of VTi2 steel after isothermal heat treatment at 898 K during 8 s.

The experimental validation of incubation time calculations has been carried out by dilatometry and metallography. The experimental incubation time is defined as the minimum time at which it is possible to detect some allotriomorphs nucleated on the austenite grain boundary. A detailed analysis of the dilatometric curve associated to the isothermal decomposition of austenite (relative change of length (dL/Lo) versus time (t)) (Fig. 1) allows to determine an interval of time, D , in which it is more likely to find the incubation time. Subsequent samples were isothermal heat treated at different holding times within the D interval. An accurate metallographic analysis of those samples determined the incubation time at which some allotriomorphs appear in the microstructure. Figure 2 shows an example of a microstructure with allotriomorphs at the initial stage of

their formation. Figure 3 shows a comparison between calculated (dashed line) and experimental (points) incubation time values for the studied steels. From this figure it can be concluded that calculated results for the incubation time are in good agreement with the predicted values from calculations proposed in this work for all the studied steels. Figure 4 presents the experimental and calculated incubation time values at the different isothermal temperatures of transformation for the three steels. Points lying on the line of unit slope show a perfect agreement between experimental and calculated values. R^2 in Fig. 4 is the square correlation factor of the experimental and calculated incubation time values. This parameter quantifies the accuracy of the calculations. An accuracy better than 90% which can be considered excellent bearing in mind the experimental difficulties for the experimental determination of the incubation time.

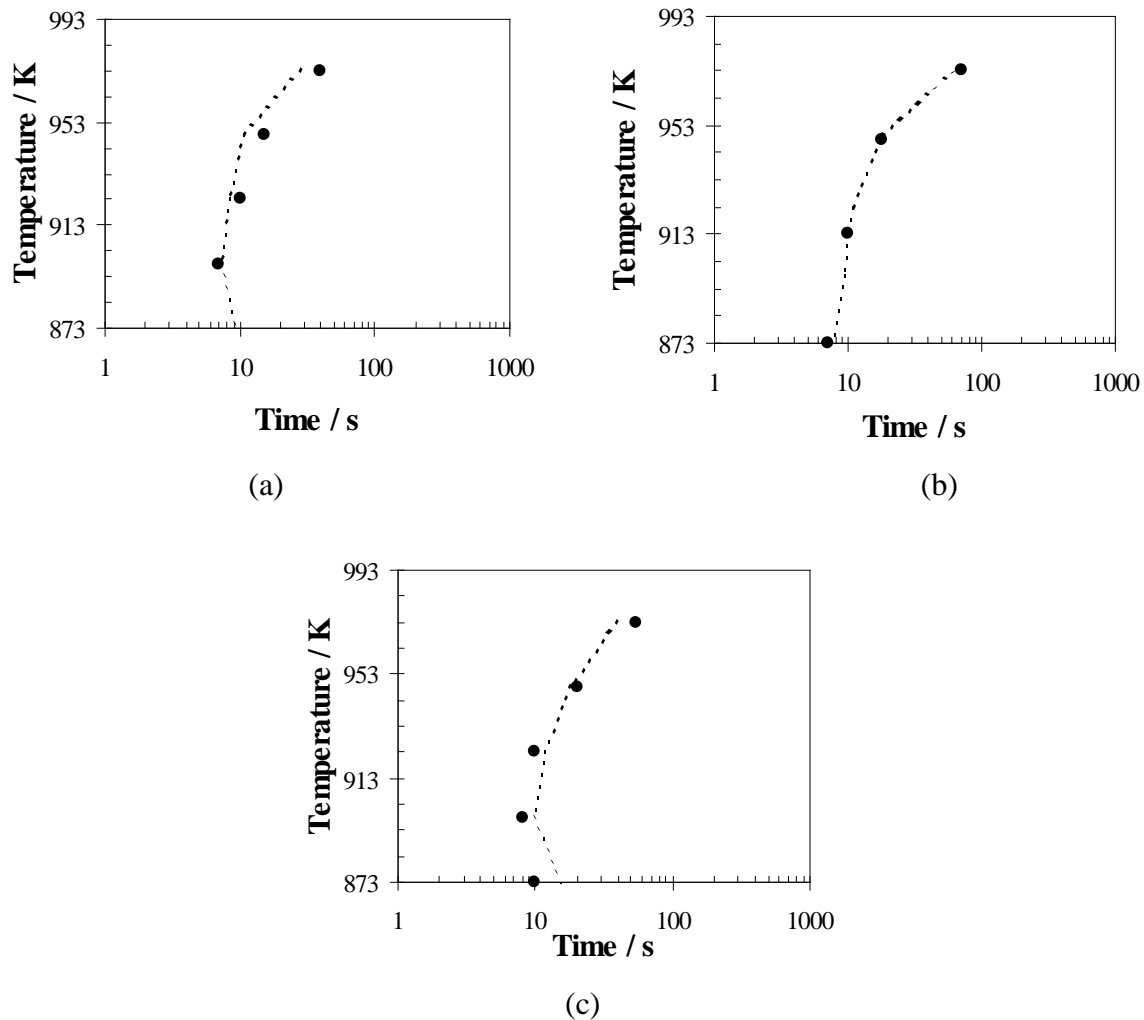


Figure 3. Experimental and calculated results for the incubation time of isothermally formed allotriomorphic ferrite in (a) CMn, (b) VTi1, and (c) VTi2 steels.

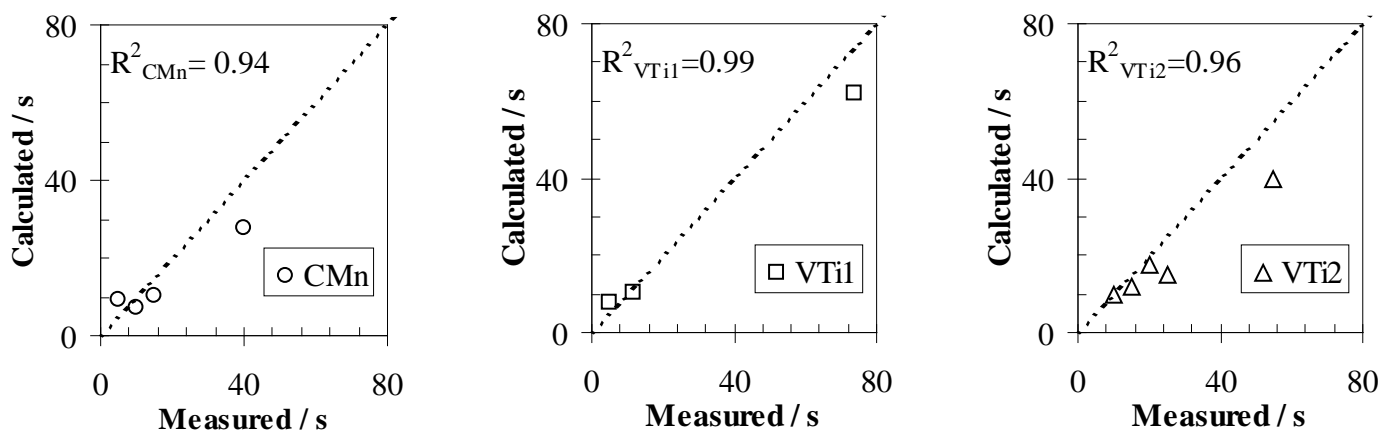


Figure 4. A comparison between calculated and experimental incubation time values in the three studied steels.

Conclusions

A mathematical process to calculate the incubation time of allotriomorphic ferrite isothermally formed in three medium carbon steels have been described. Calculations are based on well known phase transformation theories in steels. The influence of the alloying elements on the nucleation of allotriomorphic ferrite has been taken under consideration in calculations via an evaluation of their effect in the diffusivity of carbon and in the volume free energy change estimations.

Finally, experimental validation of calculations has been carried out using dilatometric and metallographic analysis. An excellent agreement (96% in R^2) between calculated and measured incubation time values for the isothermal formation of allotriomorphic ferrite has been found in the three medium carbon steels.

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