Quantitative analysis of the dilatation during an isothermal decomposition of austenites

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Abstract

The dilatation of hypoeutectoid steels during an isothermal transformation has been analysed. A model accounting for the effect of carbon redistribution has been developed which can be applied not only to the calculation of the dilatation during an isothermal transformation but also to the extraction of the phase change kinetics from a dilatation curve which may correspond to a multistep phase transformation. The model is validated by comparing it with the experimental results for an interstitial free steel. The dilatation of Fe–C steels during an isothermal transformation has been calculated and discussed. It is indicated that the isothermal formation of ferrite causes a different linear dependence of the relative length change on the volume fraction of the transformed austenite compared with the pearlite type decomposition. Isothermal transformation experiments have been carried out with an experimental bainitic grade steel. The model was successfully applied to extract the phase transformation kinetics from the dilatation curves of this steel. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Steels; Dilatation; Phase transformations; Kinetics; Mathematical model

1. Introduction

Steels exhibit a significant volume variation during heating, cooling or an isothermal transformation. As a consequence, the dilatometric technique has been employed frequently to construct time–temperature-transformation (TTT) diagrams and continuous-cooling-transformation (CCT) diagrams, which are of immense value in metallurgical applications such as heat treatment and welding, and as a means of characterising steels for particular processes. Actually, a dilatation curve records the detail of the phase transformation kinetics. Dilatometric technique may be applicable to the investigation of the phase transformation kinetics if the information contained in the dilatation curve can be extracted effectively. Works have been done in this direction. A calculation of the volume fraction of ferrite formed in a bainitic transformation in a Fe–0.3wt.%

C–4.08wt.% Cr steel, from the measured length change, was reported [1]. Later, Zwaag et al. [2] pointed out the error in this work and put forward a numerical model for hypo-eutectoid plain Fe–C steel to calculate the dilatation during an isothermal formation of primary ferrite followed by a pearlite decomposition based on the lattice parameter. The existing models are, however, not applicable to the extraction of the phase transformation kinetics from a dilatation curve. A model was, therefore, developed in the present work which could be applied not only to the calculation of the dilatation due to an isothermal transformation, but also to the extraction of the phase change kinetics from the dilatation for a multistep transformation. The model was validated by comparing with the experimental results with an interstitial free (IF) steel. The characteristics of the dilatation during an isothermal transformation of steels were discussed. Isothermal transformation experiments have been done with an experimental bainitic grade (BG) steel. The model was successfully applied to extract the phase transformation kinetics from the dilatation curves of this steel.

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2. Theoretical analyses

The density, ρ_i, and the thermal expansion coefficient, k_i^T, of a phase can be related with each other by the following equation

$$k_i^T = -\left(\frac{1}{3}\right)[\rho_i(T,C)]^{-1}\frac{\partial\rho_i(T,C)}{\partial T}, \tag{1}$$

where T is temperature, C the carbon content, subscript i refers to different phase.

Experiments have been done with pure iron to investigate the dependence of the lattice parameter of ferrite and austenite on temperature [3–10]. It has been demonstrated that the thermal expansion coefficient of austenite is approximately constant up to 1200 °C [4,5,10], and that ferrite expands also approximately linearly with temperature above 300 °C [5,6,10]. The linear dependence of lattice parameter on temperature may hold even for steels with addition of alloying element [4,10]. In fact, it is a common phenomenon that a steel contracts linearly with decreasing temperature if there is no phase transformation taking place during a cooling above 300 °C. Assuming the linear expansion with temperature holds in the temperature range 300–1200 °C for both ferrite and austenite of a given composition, their densities may be obtained by integrating Eq. (1)

$$\rho_i(T,C) = \rho_i(T_a,C)e^{-3k_i^T(T-T_0)}, \tag{2}$$

where T_a is an arbitrarily selected temperature between 300 and 1200 °C.

Works on the effect of carbon content on the lattice parameter of austenite [11,12] and ferrite [13] showed that their densities vary linearly with the carbon content for a constant temperature. According to these researches, density $\rho_i(T_a,C)$ can be given as [14]

$$\rho_i(T_a,C) = \rho_i(T_a,C=0)g_i(C), \tag{3}$$

where $g_\gamma(C) = 1 - 0.0146\% \; C$ for austenite and $g_\alpha(C) = 1 - 0.0262\% \; C$ for ferrite.

For an isothermal transformation, the relative length change is determined by

$$\frac{\partial\varepsilon}{\partial t} = -k^{\text{Trans}}(t)\frac{\partial f_\gamma}{\partial t} + k_\gamma^C f_\gamma(t)\frac{\partial C_\gamma}{\partial t}, \tag{4}$$

$$k_\gamma^C = -\frac{1}{3}\rho_\gamma^{-1}\frac{\partial\rho_\gamma}{\partial C_\gamma} = -\frac{1}{3}\frac{g_\gamma'(C_\gamma)}{g_\gamma(C_\gamma)}, \tag{5}$$

where $\varepsilon = (L-L_0)/L_0$ is the relative length change; $f_\gamma(t)$, the volume fraction of the residual austenite; k^{Trans}, the transformation expansion coefficient; k_γ^C, the solute expansion coefficient; and L_0, the length of the sample at the reference temperature. In the right hand of this equation, the first term accounts for the dilatation due to the phase transformation and the second term that due to the enrichment of carbon in the residual austenite.

The phase transformations during isothermal holding of a hypo-eutectoid austenite may be divided into three groups: (1) the precipitation type transformation which includes the formation of primary ferrite and the formation of bainitic ferrite during the first period of an upper bainitic transformation; (2) the pearlite type transformation which includes the pearlite decomposition and the last period of an upper bainitic phase transformation when the carbon-enriched residual austenite transforms into a mixture of bainitic ferrite and cementite (the expansion may be calculated just as if the austenite decomposed into a mixture of bainitic ferrite and cementite, although in reality the cementite precipitates form the austenite); and (3) the martensitic transformation which does not involve any redistribution of alloying elements, including carbon. The martensitic transformation will not be discussed in this paper, because it takes place very often below 300 °C, where the ferrite does not expands linearly with temperature. The lower bainitic transformation is not mentioned because of the uncertainty about the redistribution of carbon in this transformation. The lower bainitic transformation is being investigated using the dilatometric technique with the help of the model developed here and the results will be reported elsewhere.

For the formation of the primary ferrite and the bainitic ferrite in an austenite, it is imagined that a transformation completes in two steps: First an imagined small region of austenite adjusts its carbon content to $C_{x\alpha}$, which equals the equilibrium carbon content of ferrite, $C_{P\alpha}$, for the formation of the primary ferrite, and the carbon content of the bainitic ferrite, $C_{B\alpha}$, for the formation of the bainitic ferrite, through the rejection of the excess carbon $C_\gamma - C_{x\alpha}$. Then this small region transforms to ferrite. Therefore, $k_{\gamma\to x\alpha}^{\text{Trans}}$ and C_γ may be given by

$$k_{\gamma\to x\alpha}^{\text{Trans}} = k_{\gamma\to x\alpha}^{\text{Trans}'} + k_\gamma^C(C_{x\alpha} - C_\gamma), \tag{6}$$

$$k_{\gamma\to\alpha}^{Trans'} = \sqrt[3]{\frac{\rho_\gamma(T_a,C=0)}{\rho_{x\alpha}(T_a,C=0)}} \times \sqrt[3]{\frac{e^{-3k_\gamma^T(T_a-T)}g_\gamma(C_{x\alpha})}{e^{-3k_\alpha^T(T_a-T)}g_\alpha(C_{x\alpha})}} - 1, \tag{7}$$

$$\frac{\partial C_\gamma}{\partial t} = -(C_\gamma - C_{x\alpha})\frac{1}{f_\gamma(t)}\frac{\partial f_\gamma}{\partial t}, \tag{8}$$

where the subscript Pα and Bα indicate the primary ferrite and bainitic ferrite, respectively.

It should be mentioned that the enrichment of the residual austenite with carbon starts at the transformation interface. This may lead to a carbon concentration profile across the austenite grain. However, as far as, we use the two step transformation assumption described above for the detail of the phase transformation, the distribution of carbon in a grain does not affect $k_{\gamma\to\alpha}^{\text{Trans}'}$. Also, because $g_\gamma(C)$ is a linear function of carbon content and, in addition, is very close to unity,

Table 1
Chemical composition of the IF steel and the experimental BG steel (wt.%)

	C	Si	Mn	P	S (ppm)	N (ppm)	Al	Ti	Nb	B	Cr	Ni
IF	0.0034	0.007	0.97	0.073	38	27	0.043	0.0	0.025	0.001		
BG	0.155	0.024	1.75	0.0128	15	20	0.041	0.0821	0.106		0.023	0.023

the inhomogeneity in carbon distribution in grains has only a negligible effect on k_γ^C. So instead of calculating the carbon concentration profile across the grain, we calculate with the average concentration of carbon in austenite.

For a pearlite type transformation, it may be reasonable to assume that the austenite decomposes into two phases without carbon redistribution. The mass fraction of ferrite in the two-phase mixture, a_1, depends on the isothermal temperature. It is determinable by using the phase diagram based on carbon conservation relation. The density of the mixture of ferrite and cementite ρ_{Mix} may be calculated by [14]

$$\rho_{Mix} = \frac{\rho_{x\alpha}\rho_{Fe_3C}}{a_1\rho_{Fe_3C} + (1-a_1)\rho_{x\alpha}}, \tag{9}$$

where the subscript x is Pearlite for the pearlite decomposition or B for the bainitic transformation.

The expansion coefficient is

$$k_{\gamma \to Mix}^{Trans} = \sqrt[3]{\frac{\rho_\gamma(T,C_\gamma)}{\rho_{Mix}(T,C_\gamma)}} - 1. \tag{10}$$

Eq. (4) was solved numerically. The model can be applied not only to the calculation of the dilatation during an isothermal transformation but also to the extraction of phase transformation kinetics from a dilatation curve. The parameters needed for calculating the phase transformation kinetics from a dilatation curve are the thermal expansion coefficient k_i^T and the density $\rho_i(T_a,C=0)$. Generally k_i^T is relatively easy to be determined from the dilatation curves. The key for the calculation is the determination of $\rho_i(T_a,C=0)$. It should be mentioned that $\rho_i(T_a,C=0)$ is essentially the true density of the phase. The calculation of it is difficult especially because generally there are defects, i.e., dislocations etc., in steels. In fact, the density functions constructed are used only for the calculation of the dilatation due to the phase transformations. Eq. (7) and Eq. (10) indicate that this dilatation depends mainly on the ratio $\rho_\gamma(T_a,C=0)/\rho_{x\alpha}(T_a,C=0)$. It has been proved that a few kg m^{-3} difference in $\rho_\gamma(T_a,C=0)$ does not really affect the calculation results if only this ratio is selected properly. It may, therefore, be practical to calculate the density $\rho_\gamma(T_a,C=0)$ using the lattice parameter and take the density $\rho_{x\alpha}(T_a,C=0)$ as the value that allows the best match between the model and the experiments. For a given steel, once a single parameter set is selected correctly, it can be applied to

the extraction of the phase transformation kinetics from dilatation curves of significantly different isothermal temperatures. The whole procedure will be demonstrated in the followed Section 5.

3. Verification of the model

Experiments were carried out with an IF steel to validate the model developed. The chemical composition of the steel is given in Table 1. The steel was heated to 1230 °C, held temperature for 2 min and then quenched to 703 °C. The dilatation curve is shown in Fig. 1.

The dilatation was also calculated with this model. The thermal expansion coefficients for ferrite and austenite were taken as 1.61×10^{-5} and 2.18×10^{-5} 1/°C according to the dilatation curve. The temperature T_a was taken as 727 °C. The lattice parameter of ferrite and austenite for pure iron at T_a is 0.28965 and 0.36309 nm, respectively [10]. The effect of the main alloying element Mn on the lattice parameter was taken into account by using Eq. (11a) for the austenite [15] and Eq. (11b) for the ferrite [16]

$$a_\gamma = 3.6309(1 + 2.7 \times 10^{-4} \text{ at.\% Mn}), \tag{11a}$$

$$a_\alpha = 2.8965 + 0.00067 \times \text{at.\% Mn}. \tag{11b}$$

The densities $\rho_\gamma(T_a,C=0)$ and $\rho_{P\alpha}(T_a,C=0)$ calculated from the lattice parameters are 7735 and 7625 kg m^{-3}, respectively. The composition of the steel and the isothermal holding temperature determine that there is
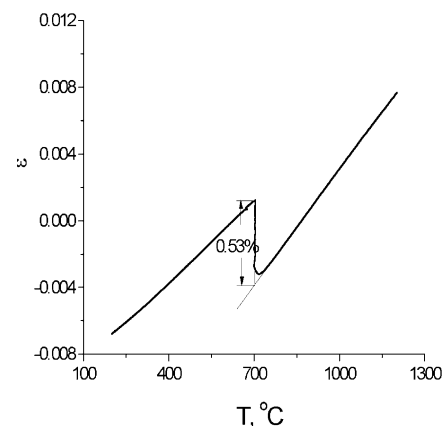

Fig. 1. Dilatation during an isothermal transformation of the IF steel.
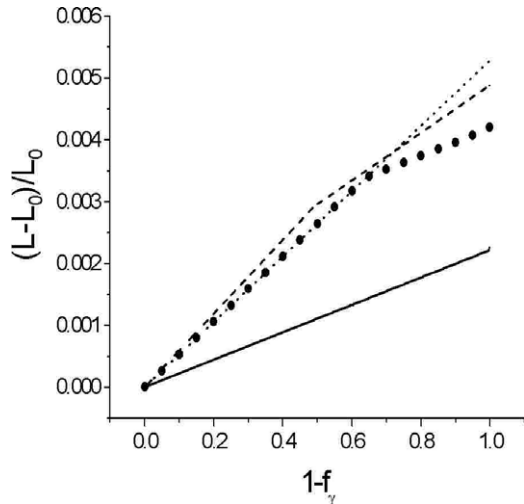
Fig. 2. Dilatation during an isothermal decomposition of austenite. Dots, circles and solid line are for Fe–0.02wt.% C, Fe–0.3wt.% C and Fe–0.8wt.% C steels transformed at 723 °C, dashes are for Fe–0.3wt.% C steel transformed at 600 °C.
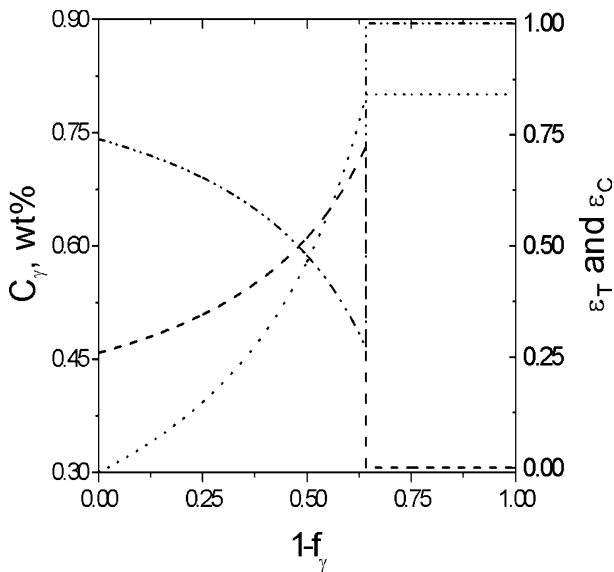

Fig. 3. The variation of the carbon content in the residual austenite (dots), the instantaneous relative contribution of the dilatation due to phase transformation, $\varepsilon_T = -k^{\mathrm{Trans}}(t)\partial f_\gamma/\partial\varepsilon$ (dash dots), and/or due to the carbon redistribution, $\varepsilon_C = k_\gamma^C f_\gamma \partial C_\gamma/\partial\varepsilon$ (dashes), during an isothermal transformation of Fe–0.3wt.% C steel

4. Numerical results of the dilatation on phase transformation and discussions

The dilatation of Fe–C steels during an isothermal transformation was calculated. Supposing the austenite of Fe–0.3wt.% C steel is suddenly quenched to 723 °C and held there, Fig. 2 shows the dilatation as a function of the volume fraction of the transformed austenite. It indicates that the primary ferrite forms first. This leads to the enrichment of carbon in the residual austenite, as shown in Fig. 3. The enrichment of carbon is accelerated and, therefore, its relevant contribution to the dilatation is enhanced with the progress of the transformation, just as predicted by Eq. (8). This phenomenon continues until the residual austenite is saturated with carbon and tends to decompose into a two-phase mixture of ferrite and cementite, Fig. 3, owning to the subsequent pearlite transformation. Both the formation of the primary ferrite and the followed pearlite decomposition cause a linear dilatation with the amount of the transformed austenite. Compared with the formation of ferrite, pearlite decomposition has a much lower dilatation slope. Calculations for steels of Fe–(0.02–0.8)wt.% C transformed at 723 °C showed that the carbon content does not affect the slope of the dilatation curve for the formation of the primary ferrite or pearlite decomposition, but only the relevant amount of pearlite (Fig. 2). These phenomena are understandable. In fact, for an isothermal phase transformation during which carbon does not redistribute, the dilatation should obviously be a linear function of the transformed austenite. The constant slope of the dilatation curve equals the transformation expansion coefficient given by Eq. (7) for the formation of the primary ferrite or by Eq. (10) for a pearlite decomposition. Further, because the carbon effect on the density of austenite is separable from the temperature effect and, in addition, it is a linear function of carbon content and is very close to unity, k_γ^C determined by Eq. (5) is almost a constant. Therefore, for a transformation during which carbon redistribution takes place, when the residual austenite receives carbon atoms and expands, the imagined small region of austenite which transforms to new phase gives out carbon atoms and contracts almost the same amount. That is, the carbon redistribution in austenite does not change the linear relation between the dilatation and the volume fraction of the transformed austenite. The slope of the dilatation curve is still $k_{\gamma\to\alpha}^{\mathrm{Trans'}}$. This can be seen clearly by substituting Eq. (6) and Eq. (8) into Eq. (4).

The isothermal temperature may affect the slope of the dilatation curve corresponding to the pearlite type decomposition, $k_{\gamma\to\mathrm{Mix}}^{\mathrm{Trans}}$, because, on the one hand, it determines the carbon content of the austenite at

only one phase transformation. The carbon does not redistribute during the transformation. It can be seen by integrating Eq. (4) that the total relative length change equals $k_{\gamma\to\alpha}^{\mathrm{Trans'}}$. Calculation showed that it is 0.53%. It coincides exactly with the experimental value (Fig. 1).

which the decomposition takes place, and on the other hand, the density of ferrite and austenite decreases with temperature at different rates due to the difference between their thermal expansion coefficients. The latter also causes an increasing slope of the dilatation curve corresponding to the formation of the primary ferrite, $k_{\gamma \to \alpha}^{\mathrm{Trans'}}$, with decreasing temperature (Fig. 2). The analyses above indicate that if an isothermal process involving only one phase transformation, the transformed amount of austenite at any time can be calculated from the dilatation curve by using the single linear relationship between the relative length change and the transformed amount of austenite. This rule is not applicable to an isothermal process where the final reaction product is formed in separate, consecutive decompositions. This seems still not to be fully realized [17].
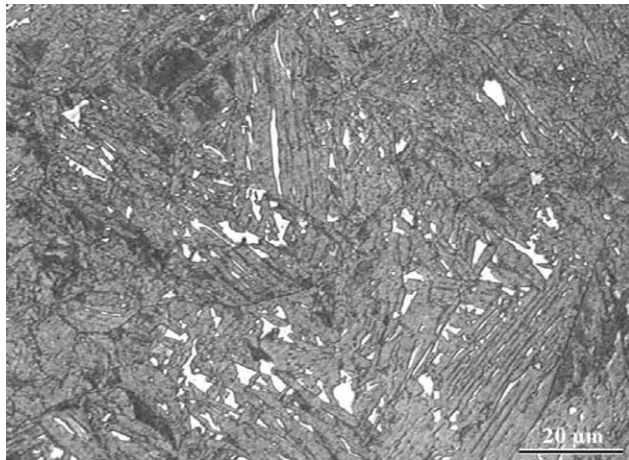
(a)
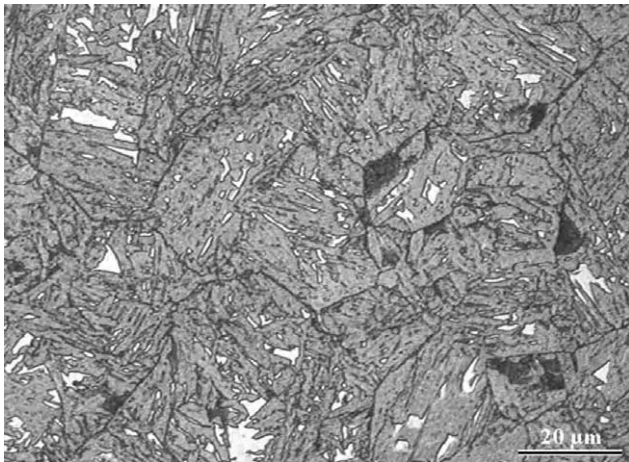
(b)

Fig. 4. Optical micrographs showing the microstructure of the experimental BG steel after the isothermal holding at (a) 548 °C and (b) 528 °C. The bright white phase is the martensite, the black phase is the mixture of bainitic ferrite and cementite.

5. Extraction of the phase change kinetics from dilatation curves for a multistep isothermal transformation

Experiments were done with an experimental BG steel. The chemical composition of the steel is given in Table 1. The steel was heated to 1200 °C, held at this temperature for 2 min and then quenched at a rate of 3000 °C min^{-1} to a different temperature in the range 450–550 °C. The steel was further quenched to room temperature after 45–60 min isothermal holding. The microstructure examination indicated that the steel was consisted of bainite and martensite (Fig. 4). The carbon content of the martensite, which equals the carbon content of the austenite just before the sample was quenched to room temperature, was measured. It was demonstrated that the bainitic transformation start temperature, B_s, can be predicted by Ref. [18]

$$B_s(°C) = 830 - 270 \mathrm{wt.\%\ C} - 90\mathrm{wt.\%}$$

$$\mathrm{Mn} - 37\mathrm{wt.\%\ Ni} - 70\mathrm{wt.\%\ Cr} - 83\mathrm{wt.\%\ Mo}. \qquad (12)$$

Experiments showed that the thermal expansion coefficient for the austenite of this steel is 2.25×10^{-5} 1/°C, whereas that for the bainitic ferrite is 1.48×10^{-5} 1/°C. The density of cementite can be calculated by Ref. [14]

$$\rho_{\mathrm{Fe_3C}}(T \text{ in } °C)$$

$$= 7686.45 - 6.63 \times 10^{-2}T - 3.12 \times 10^{-4}T^2. \qquad (13)$$

The temperature T_a was taken as 727 °C. The lattice parameter of the austenite at T_a was calculated by Ref. [15]

$$a_\gamma = 3.6309(1 + 2.7 \times 10^{-4}\mathrm{at.\%\ Mn} + 7.8$$

$$\times 10^{-4}\mathrm{at.\%\ Al} + 2.21 \times 10^{-3}\mathrm{at.\%\ Nb} + 1.7$$

$$\times 10^{-4}\mathrm{at.\%\ Cr} - 5.6 \times 10^{-5}\mathrm{at.\%\ Ni} + 8.9$$

$$\times 10^{-4}\mathrm{at.\%\ Ti}). \qquad (14)$$

The density $\rho_\gamma(T_a, C = 0)$, as calculated from the lattice parameters, is 7725 kg m^{-3}. The density of the bainitic ferrite $\rho_{\mathrm{B}\alpha}(T_a, C = 0)$ was taken as 7635 kg m^{-3}, the value which allowed the best match between the practical phase constituents of samples and the ones predicted by the model.

Calculations were carried out for the samples transformed at 528 and 548 °C. The dilatation of the samples is shown in Fig. 5 as a function of temperature. The calculated phase transformation processes from the dilatation curves are shown in Fig. 6 together with the dilatation as a function of time. Further the dilatation is shown in Fig. 7 together with the carbon content of the residual austenite as a function of the volume fraction of the transformed austenite. Figs. 6 and 7 demonstrate that the bainitic ferrite forms first.
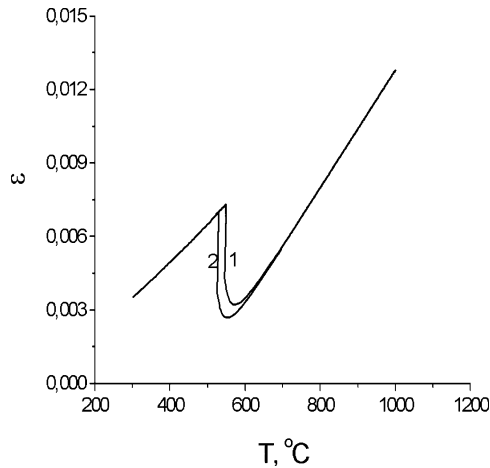
Fig. 5. The relative length change of the sample transformed at 548 °C (line 1) and 528 °C (line 2).
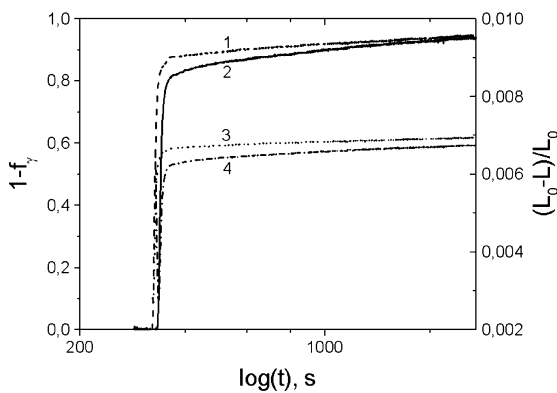

Fig. 6. The volume fraction of the transformed austenite (line 1: transformed at 528 °C; line 2: transformed at 548 °C) and the relative length change of the samples (line 3: transformed at 528 °C; line 4: transformed at 548 °C).

This transformation causes an enrichment of carbon in the residual austenite. The residual austenite transforms into a mixture of bainitic ferrite and cementite, when the carbon content of the residual austenite reaches B_s line. The calculations gave a 95% transformation of the austenite during the isothermal holding for both samples. It coincides very well with the metallographic examinations (Fig. 4). Fig. 7 demonstrates further that the formation of the bainitic ferrite causes a different linear dependence of the dilatation on the volume fraction of the transformed austenite as the late stage decomposition of the austenite when its carbon content reaches the B_s does, as discussed in Section 4.

6. Conclusions

A detailed analysis has been made of the relative length change of a hypoeutectoid steel during an isothermal transformation. A model has been devel-
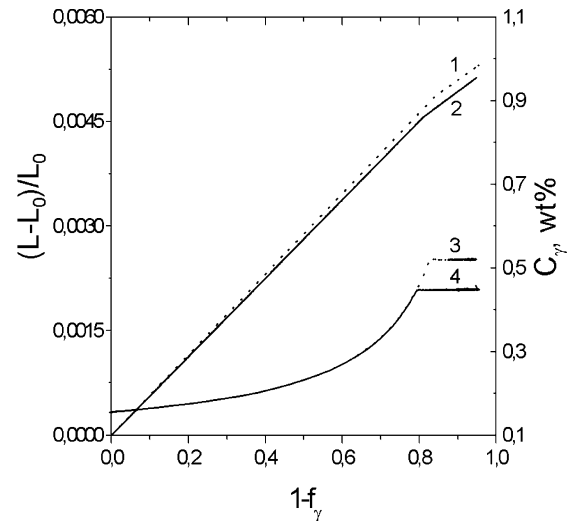

Fig. 7. The relative length change (line 1: transformed at 528 °C; line 2: transformed at 548 °C) and the carbon content of the residual austenite (line 3: transformed at 528 °C; line 4: transformed at 548 °C,) versus the volume fraction of the transformed austenite during an isothermal transformation.

oped which takes into account the effect of carbon segregation on the dilatation and which can be applied, not only to the calculation of the dilatation during an isothermal transformation, but also to the extraction of the phase change kinetics from a dilatation curve which may correspond to a multistep phase transformation. The model was validated by comparing it with the experimental results for an IF steel. The dilatation of Fe–C steels during an isothermal transformation has been calculated and discussed. It has been indicated that the isothermal formation of ferrite causes a different linear dependence of the relative length change on the volume fraction of the transformed austenite as the pearlite type decomposition does. Isothermal transformation experiments have been done with an experimental BG steel. The model has been successfully applied to the extraction the phase transformation kinetics from the dilatation curves of this steel.

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