Jarosław OPARA, Andrzej WROŻYNA

Instytut Metalurgii Żelaza

DEVELOPMENT AND VALIDATION OF A QUANTITATIVE DILATOMETRIC ANALYSIS MODEL OF AUSTENITE DECOMPOSITION INTO FERRITE AND PEARLITE

The quantitative dilatometric analysis model for the austenite decomposition into ferrite and pearlite during continuous cooling treatment is presented. It is based on measurements of a relative length change of a sample during the progress of phase transformations and calculations of the difference in atomic volume of present phases. The proposed model incorporates crucial effects accompanying the austenite decomposition, i.e. the carbon enrichment of the remaining austenite during ferrite formation, which causes an increase in the definite atomic volume of austenite, while formation of pearlite has a clearly different impact with the volume fractions of cementite and ferrite regulated by constant carbon composition inherited from austenite (enriched in carbon). The analysis results are compared with quantitative microstructure analysis and an excellent convergence has been found. Depicted results state a convincing confirmation that the model is correctly developed by the authors. Additionally, kinetics of phase transformations as function of normalized time were analyzed and a process of carbon enrichment of austenite was demonstrated.

Key words: hypoeutectoid steel, dilatometry, austenite decomposition, ferrite, pearlite, lattice parameter, kinetics

OPRACOWANIE I WALIDACJA MODELU ILOŚCIOWEJ ANALIZY DYLATOMETRYCZNEJ ROZPADU AUSTENITU W FERRYT I PERLIT

W niniejszej pracy zaprezentowano model ilościowej analizy dylatometrycznej dla rozpadu austenitu w ferryt i perlit podczas ciągłego chłodzenia. Rozwiązanie bazuje na pomiarach względnej zmiany długości próbki w trakcie postępu przemian fazowych oraz obliczeniach różnic objętości atomowych występujących faz. Zaproponowany model uwzględnia kluczowe zjawiska towarzyszące rozpadowi austenitu, tzn. wzbogacanie się austenitu w węgiel podczas powstawania ferrytu, które powoduje wzrost określonej objętości atomowej austenitu, podczas gdy formowanie się perlitu ma wyraźnie odmienny wpływ poprzez udziały objętościowe ferrytu i cementu, które wynikają z ustalonej zawartości węgla dziedziczonej z austenitu (wzbogaconego w węgiel). Rezultaty ilościowej analizy dylatogramów zestawiono z ilościową analizą mikrostruktur i uzyskano doskonałą zbieżność wyników. Przedstawione wyniki stanowią wyraźne potwierdzenie, iż model jest poprawnie opracowany przez autorów. Ponadto, przeprowadzono analizę kinetyk przemian fazowych w funkcji znormalizowanego czasu oraz przedstawiono proces wzbogacania austenitu w węgiel.

Stowa kluczowe: stal podeutektoidalna, dylatometria, rozpad austenitu, ferryt, perlit, parametr sieci, kinetyka

1. INTRODUCTION

The formation of ferrite and pearlite during austenite decomposition is a process with a considerable technological significance, for example in production of hotforged steel forgings with desirable yield strength (in BY heat treatment). One of the well known methods to study phase transformations in steels is a dilatometric technique. In the literature one can find several works [1–8] associated with dilatometric analysis to quantify the volume fraction of transformation products. These works incorporate crucial effects accompanying the austenite decomposition, i.e. the carbon enrichment of the remaining austenite during ferrite formation, which causes an increase in the definite atomic volume of austenite, while formation of pearlite has a clearly different impact with the volume fractions of cementite and ferrite regulated by constant carbon composition inherited from austenite (enriched in carbon). Nevertheless, none of these approaches has been available,

i.e. to common use of open access source code or a computer application. Hence, there was a motivation to develop its own model based on the previous works [4, 5, 7]. Therefore, one of the objectives of the work was reconstruction of the dilatometric analysis approach proposed by Kop et al. [4]. Accordingly, a computer program was developed with an implemented quantitative dilatometric analysis model, and with additional features to deal with dilatometric data.

Another purpose of this work was to evaluate and validate the proposed solution. The validation was performed using the data from the work of Kop et al. [4], and experiments carried out for a low carbon steel. An application of the model on continuous cooling experiments is demonstrated. The analysis results are compared with metallographic measurements using an image analysis technique and an excellent convergence has been found. The results are depicted in chapters 4 and 6.

2. THEORY

Dilatometry is based on measurements of a relative volume change of a sample during progress of the phase transformations driven by the specified thermal cycle. The relative volume change of the material results from both the thermal expansion/contraction effect and the dimensional changes associated with the phase transformation phenomena at atomistic scale, i.e. a change in the crystal structures and/or an enrichment/depletion of the solute in solid solutions. The total relative volume change can be described by semiempirical models to quantify the progress of phase transformations, what is extensively demonstrated in the literature [1-8]. This approach allows to perform so called quantitative dilatometric analysis of a particular dilatation curve. For the proposed solution the following assumptions need to be adopted: transformation strains are neglected and the expansion/contraction of a sample is isotropic. Under the above assumptions, for small dimensional changes, the following dependence of the relative total atomic volume change regarding the relative length change can be defined [2, 4, 5]

$$\frac{\Delta V}{V_0} = \left(1 + \frac{\Delta L}{L_0}\right)^3 - 1 \approx \frac{3\Delta L}{L_0} \tag{1}$$

where: $\Delta V = V - V_0$ is the atomic volume change equal to difference between total average atomic volume V at a certain temperature and the initial average atomic volume V_0 at the reference temperature; $\Delta L = L - L_0$ – the measured length change expressed by the difference between the specimen length L at a certain temperature and the initial length L_0 at room temperature.

The right-hand side term in the equation (1) is the value directly obtained from the dilatation measurements, while the left-hand side term can be calculated based on the atomic volumes of the existing phases. The average atomic volume of the alloy is directly related to the volume fractions of structural constituents present in the material at a certain temperature T and can be formulated with the volume fraction rule

$$V(T) = \sum_{i} f_i V_i(T) \tag{2}$$

where: V is the average atomic volume of the sample; f_i – the volume fraction of a structural constituent i; V_i – the atomic volume of the structural constituent i.

Generally, in hypoeutectoid steels below A_3 temperature we can consider three phases, i.e. austenite, ferrite and cementite. Therefore, the following equations to determine atomic volumes of the mentioned phases, which are related to the lattice parameters, are presented respectively

$$V_{\gamma} = \frac{1}{4}a_{\gamma}^{3} \tag{3}$$

$$V_{\alpha} = \frac{1}{2} a_{\alpha}^{3} \tag{4}$$

$$V_{\theta} = \frac{1}{12} a_{\theta} b_{\theta} c_{\theta} \tag{5}$$

where: *a* is the lattice parameter of austenite, ferrite and cementite phase for the following subscripts γ , α , θ , respectively; b_{θ} and c_{θ} are the lattice parameters of the cementite phase in the other two spatial coordinates. The factors 4, 2 and 12 in the denominator resulting from the fact that 4, 2 and 12 iron atoms are contained in the unit cells of austenite, ferrite and cementite, respectively.

Additionally, during cooling of hypoeutectoid steels below A_1 temperature there appear phase mixtures of ferrite and cementite, i.e. structural constituent pearlite for which atomic volume can be calculated from the following formulae

$$V_p = (1 - \rho)V_{\alpha} + \rho V_{\theta} \tag{6}$$

where ρ is the fraction cementite in the pearlite.

The lattice parameters of the presented phases in the equations $(3\div5)$ can be determined using a general equation

$$a_{\phi} = (a_{\phi}^{0} + w_{\phi} x_{\gamma}^{C}) [1 + \beta_{\phi} (T - T_{0})]$$
(7)

where: a_{ϕ}^{0} is the lattice parameter in unalloyed phase at the reference temperature T_{0} at which the lattice parameter was determined; w_{ϕ} – the coefficient relating the effect of the carbon concentration to the phase lattice parameter; x_{γ}^{C} – the average carbon concentration in austenite; β_{ϕ} – the linear thermal expansion/ contraction coefficient of the certain phase; ϕ – the identifier of the considered phases with the following symbols γ , α , θ for austenite, ferrite and cementite, respectively. The lattice parameters used in the developed model are listed in Tab. 1.

The linear thermal expansion/contraction coefficients for austenite and ferrite are defined as a linear function of carbon concentration

$$\beta_{\phi} = e_{\phi} - k_{\phi} x_{\gamma}^{C} \tag{8}$$

where: e_{ϕ} is the constant parameter; k_{ϕ} – the coefficient relating the effect of the carbon concentration to the thermal expansion/contraction coefficient.

Unlike the previous approach, the linear thermal expansion/contraction coefficient for cementite is described by square function of temperature

$$\beta_{\theta} = e_{\theta} - g_{\theta}T + h_{\theta}T^2 \tag{9}$$

where: g_{θ} and h_{θ} are the coefficients relating the effect of the temperature to the thermal expansion/contraction coefficient of cementite.

Basing on the equations $(1\div9)$ it is not possible to calculate the phase fractions from the volume change of a sample due to the two intrinsic factors that prevent their direct determination, namely: the simultaneous

Table 1. Published data for lattice parameters of austenite, ferrite and cementite as function of composition and temperature from reference [1, 9]

Tabela 1. Opublikowane dane parametrów sieci austenitu, ferrytu i cementytu w funkcji składu chemicznego oraz temperatury ze źródeł [1, 9]

Phase	a_{ϕ}^{0}	b_{ϕ}^{0}	c_{ϕ}^{0}	w _φ	e_{ϕ}	k_{ϕ}	g_{ϕ}	h_{ϕ}	T_0
ϕ	[nm]	[nm]	[nm]	[nm/at.%]	[1/K]	[1/K /at.%]	[1/K]	$[1/K^2]$	[K]
Austenite, γ	0.36306	0.0	0.0	$7.8 \cdot 10^{-5}$	$2.49 \cdot 10^{-5}$	$5.0 \cdot 10^{-7}$	0.0	0.0	1000.0
Ferrite, α	0.28863	0.0	0.0	0.0	$1.75 \cdot 10^{-5}$	0.0	0.0	0.0	800.0
Cementite, θ	0.45234	0.50883	0.67426	0.0	$5.311 \cdot 10^{-6}$	0.0	$1.942{\cdot}10^{-9}$	$9.655 \cdot 10^{-12}$	293.0

25

formation of the two phases, i.e. ferrite and pearlite, and the carbon-concentration dependence of the atomic volume of austenite [4]. The first factor stems from the fact that the dilatation curve cannot distinguish between the formation of pro-eutectoid ferrite and pearlite. Therefore, certain arbitrary assumptions need to be taken to define separate temperature regions where the formation of ferrite and pearlite takes place, as it is expected from the phase diagram. Hence, the dilatation curve can be analyzed in two steps. The problem with unambiguous determination of the atomic volume of austenite can be solved using the variation of the lattice parameters with the temperature and composition determined by Onink et al. [1, 9] and mass conservation relation. Considering the two products of the austenite decomposition, i.e. ferrite and pearlite, the following equation for the mass conservation relation, based on the volume fraction rule, can be expressed

$$x_0^C = f_\gamma x_\gamma^C + f_\alpha x_\alpha^C + f_p x_p^C \tag{10}$$

where: x_0^C – is the average carbon concentration in the material; x_{α}^C – is the carbon concentration in ferrite under the thermodynamic condition; x_p^C – is the carbon concentration in pearlite.

Furthermore, during the progress of the austenite to ferrite phase transformation a carbon enrichment of the austenite occurs due to constrained solubility of carbon in ferrite, which depends on the momentary ferrite fraction. Accordingly, the average carbon content of the austenite, x_{γ}^{C} , in a certain stage of the austenite decomposition into ferrite (at this stage of transformation there is no pearlite, i.e. $f_{p} = 0$), can be calculated from a reconstructed equation (10) as follows

$$x_{\gamma}^{C} = \frac{x_{0}^{C} - f_{\alpha}x_{\alpha}^{C}}{f_{\gamma}} = \frac{x_{0}^{C} - f_{\alpha}x_{\alpha}^{C}}{1 - f_{\alpha}}$$
(11)

Summarizing, the volume fraction of the phases can be determined from the dilatation curve by utilizing the lattice parameters, the linear thermal expansion coefficients and the phase compositions under the thermodynamic condition. However, on the account of the complexity of the problem an analytical solution for the equations (1) and (2) does not exist. Therefore, iterative procedures and numerical methods are developed with above assumptions and equations $(1\div11)$ as a computer program. The proposed model and its implementation is described in next section.

3. MODEL DEVELOPMENT

The quantitative dilatometric analysis model for the austenite decomposition into ferrite and pearlite during a continuous cooling treatment is proposed. Accordingly, and based on the equation (2) the following equation to determine the total atomic volume, V, of the alloy can be deduced

$$V = f_{\gamma} V_{\gamma +} f_{\alpha} V_{\alpha +} f_{p} V_{p} \tag{12}$$

where: f – is the volume fraction of austenite, ferrite and pearlite for the following subscripts γ , a, p, respectively.

The dilatometric analysis is performed separately in two steps by the assumption that the austenite decomposition is divided into two separate temperature regions, where the formation of ferrite and pearlite appears, as is expected from the phase diagram. Therefore, the crucial aspect of the analysis is to define a characteristic temperature, i.e. pearlite start temperature, which separates the austenite transformation into two temperature regions. There are several ways to determine this temperature, which are extensively described in the work of Kop et al. [4] and the work of Choi [6].

The suggested model is based on the carbon enrichment of the austenite and the difference in the atomic volume of phases during the phase transformation. Hence, at the first region of high temperatures when austenite decomposition starts, the volume fraction of ferrite can be calculated by the following relation

$$f_{\alpha} = \frac{V - V_{\gamma}}{V_{\alpha} - V_{\gamma}} \tag{13}$$

As it was stated earlier, there is no analytical solution for the equations (1) and (2) and consequently the equation (13). This is mainly due to the carbon enrichment of the austenite, which depends directly on the current volume fraction of ferrite at a given stage of the phase transformation. Therefore, the ferrite volume fraction is determined by an iterative scheme, in which the Newton-Raphson method [10] is used. In the iterative scheme the average carbon concentration in the austenite is calculated using the equation (11), and the carbon concentration in ferrite is determined from the phase equilibrium line in Fe-Fe₃C phase diagram which is calculated via Thermo-Calc [11]. Additionally, to solve the problem with iterative procedure the goal function needs to be defined. Accordingly, the reconstructed equation (1) with the relative change in length proportional to the ratio of atomic volumes of phases present at current temperature is applied in the following form

$$\frac{\Delta L}{L_0} = \frac{1}{3} \frac{\Delta V}{V_0} = \frac{1}{3} \frac{V - V_0}{V_0}$$
(14)

In the second temperature region only the transformation of austenite to pearlite is analyzed. Thus, in the further analysis the volume fraction of ferrite is assumed to be constant and also carbon enrichment at austenite do not occur. The pearlite composition with carbon is constant and equals to the austenite composition with the average carbon concentration immediately after finishing of the austenite to ferrite transformation. This composition determines the ratio of cementite and ferrite in the pearlite [4]. The volume fraction of pearlite can be derived directly from the equation (12) as follows

$$f_p = \frac{V - V_\gamma + f_\alpha (V_\gamma - V_\alpha)}{V_p - V_\gamma}$$
(15)

The contribution of experimental errors, i.e. some non-idealities which cause deviations between calculated and measured length change, is compensated via the scaling factor [4], κ , and the applying equation (1) is defined by

$$V = \kappa V_0 \left(\frac{3\Delta L}{L_0} + 1\right) \tag{16}$$

The scaling factor, κ , for an ideal case equals 1. Actually, always some non-idealities take place. The circumstances which influence the scaling factor and the way to determine its value are well described in the

work of Kop et al. [4]. According to this concept two reference temperatures need to be defined, i.e. before and after the austenite phase transformation, at which the two scaling factors are calculated based on the equation (16). Subsequently, a linear function with temperature is defined based on values of scaling factors. Thereby, it is possible to determine a current scaling factor in dilatometric analysis with a changing temperature.

The general scheme of the algorithm for calculating the phase transformation kinetics from dilatation data is presented in Fig. 1. This flow chart describes the data flow and a call of subsequent procedures which are necessary to obtain the final results of a quantitative dilatometric analysis, i.e. the volume fraction of transformed phases and their carbon contents.

In order to demonstrate the capabilities of the proposed model the evaluation and validation were performed based on the original data and results from the work of Kop et al. [4] as well as using experimental results carried out within this work. Obtained results are shown in the next chapters. The developed model is validated using the data from the work of Kop et al. [4]. From this publication Figure 7 was copied and digitalized with the dilatation curve of C22 steel. Prepared data (see Fig. 2), experimental information and also calculated thermodynamic data for C22 constitute an input to the analysis performed with the model developed by the authors (see Fig. 1).

The validation results are presented in Fig. 3 for the two assumptions to determine the start temperature of the austenite to pearlite transformation. For the first case it is assumed that pearlite transformation starts when a pro-eutectoid ferrite formation obtains the final equilibrium volume fraction of ferrite. The second approach is that the pearlite start temperature is determined from a dilatometric diagram based on the (second) point of inflection. For more details see reference [4].



Fig. 1. Flow chart with algorithm of a quantitative dilatometric analysis for austenite decomposition into ferrite and pearlite

Rys. 1. Schemat blokowy z algorytmem ilościowej analizy dylatometrycznej dla rozpadu austenitu w ferryt i perlit



Fig. 2. Dilatation curve during cooling (20°C/min) for C22 steel copied and digitalized from the work of Kop et al. [4] Rys. 2. Krzywa dylatacyjna podczas chłodzenia (20°C/min) stali C22, skopiowana i zdigitalizowana z pracy Kopa i in. [4]

In (Fig. 3) one can observe an excellent convergence of the calculated ferrite volume fraction as the function of temperature with digitalized data from the work [4]. However, slight discrepancies between the calculated and digitalized results can be observed for pearlite volume fraction changes with temperature, especially in Fig. 3b. This deviation can be explained either by an inaccurate digitalization of picture with dilatometric curve, from the work [4], within the pearlite transformation range or by the differences in the implementation of a procedure to calculate atomic volume of pearlite. Nevertheless, the depicted results state a convincing confirmation that the quantitative dilatometric analysis model is correctly developed by the authors. An application of the model is demonstrated in case of continuous cooling experiments of hypoeutectoid steel in the next section.

5. EXPERIMENTAL PROCEDURE

The chemical composition of steel used for dilatometric measurement is shown in Tab. 2. The dilatometric measurements were carried out on $\phi 4 \times \phi 2 \times 10.0$ mm samples using a Bähr 805A/D quench dilatometer. The samples were thermally stabilized due to heating by induction to 650°C at rate of 10°C/s, annealing for 10 minutes and cooling to room temperature at a rate of 0.5°C/s. Then the samples were heated to 920°C at a rate of 10°C/s, held for 10 minutes and subsequently cooled with different rates of 0.1°C/s, 0.5°C/s, 1°C/s, 2°C/s and 4°C/s. The characteristic temperatures of phase transformations were determined by the concept of an inflection point.

Table 2. Chemical composition of S355J2 steel (European standard steel number 1.0577) in wt.%

Tabela 2. Skład chemiczny stali S355J2 (w
g normy europejskiej numer stali 1.0577) w% wag.

С	Si	Mn	Р	S	Cu	Al	Ti	N
0.17	0.40	1.55	0.017	0.026	0.07	0.032	0.0014	0.0070

The microstructure was observed by light optical microscopy after a standard method of preparation with 3% nital etchant. Quantitative analysis of phase fractions were conducted on four images per each sample using quantitative image analysis software MetIlo [12].

6. RESULTS AND DISCUSSION

The dilatometric curves for continuous cooling at various cooling rates are shown in Fig. 4. For the analyzed values of cooling rates the two subsequent transformations are visible. The higher cooling rate value the lower start and finish temperatures and a change in the dilatation effect of the above-mentioned transformations is observed.



Fig. 3. Validation of the volume fraction change of ferrite and pearlite as a function of temperature based on the data from the work of Kop et al. [4] for C22 steel. The two assumptions are considered to determine the start temperature of austenite to pearlite transformation: (a) pearlite starts when pro-eutectoid ferrite formation obtains the final equilibrium volume fraction of ferrite; (b) pearlite start temperature is determined from a dilatometric diagram based on the (second) point of inflection

Rys. 3. Walidacja zmian udziału objętościowego ferrytu i perlitu w funkcji temperatury na podstawie danych z pracy Kopa i in. [4] dla stali C22. Przyjęto dwa założenia przy wyznaczaniu temperatury rozpoczęcia przemiany austenitu w perlit: (a) formowanie perlitu rozpoczyna się gdy przyrost ferrytu przedeutektoidalnego osiąga końcowy równowagowy udział objętości ferrytu; (b) temperatura rozpoczęcia formowania się perlitu jest określana z wykresu dylatometrycznego na podstawie (drugiego) punktu przegięcia



Fig. 4. Dilatometric curves for S355J2 steel cooled at various cooling rates. For better readability the curves are shifted along "relative change length" axis

Rys. 4. Krzywe dylatometryczne dla stali S355J2 chłodzonej z różnymi szybkościami chłodzenia. Dla zwiększenia czytelności krzywe zostały przesunięte wzdłuż osi "względnej zmiany długości"

Microstructures after cooling at a series of cooling rates are shown in Fig. 5. In all the analyzed samples a mixture of ferrite and pearlite is observed. The higher cooling rate, the smaller grain size of ferrite. The results of the quantitative image analysis for fractions of structural constituents are shown in (Tab. 3) together with the results of fraction calculations based on dilatometric measurements. The results from the developed quantitative dilatometric analysis model coincide with the quantitative image analysis ones whereas the results from the lever rule method in case of ferrite are higher on average about 10 percentage points. The fraction of ferrite is probably overestimated in the lever rule method since the carbon enrichment and the increasing atomic volume of the austenite during ferrite formation is not taken into account in the analysis of the samples dilatation.

Additionally, an application of the quantitative dilatometric analysis model gives a possibility to determine kinetics of phase transformations. The increasing volume fraction of ferrite and pearlite as a function of temperature for the analyzed experiments is presented in Fig. 6. One can observe that temperatures at which a certain fraction of ferrite and pearlite are reached decrease as the cooling rate increases. The determined values of start and finish temperatures of transformations taken into consideration are presented in Tab. 4. A temperature span between start and finish of pearlitic transformation (Tab. 4) is the highest at 1°C/s and 2°C/s, where it is equal $\Delta T = 50$ °C and both for higher and lower cooling rates decreases to circa $\Delta T = 34^{\circ}$ C. And in case of ferritic transformation the temperature span remains at around $\Delta T = 118$ °C.

Table 3. Volume fractions of ferrite and pearlite in steel S355J2 cooled at various cooling rates determined in the quantitative image analysis and on the basis of the length change of the samples by the lever rule method and the developed quantitative dilatometric analysis model

Tabela 3. Ułamki objętości ferrytu i perlitu w stali S355J2 chłodzonej z różnymi szybkościami chłodzenia wyznaczone na podstawie ilościowej analizy obrazu oraz reguły dźwigni bazującej na zmianach długości próbki oraz opracowanego modelu ilościowej analizy dylatogramów

Cooling rate	Quantitative i	mage analysis	Leve	r rule	Quantitative dilatometric analysis model	
	Ferrite	Pearlite	Ferrite	Pearlite	Ferrite	Pearlite
[°C/s]	[%]	[%]	[%]	[%]	[%]	[%]
0.1	71.4	28.6	80.2	19.8	70.9	29.1
0.5	73.7	26.3	82.9	17.1	72.6	27.4
1	76.8	23.2	84.1	15.9	73.9	26.1
2	77.2	22.8	88.0	12.0	75.2	24.8
4	73.9	26.1	89.3	10.7	76.7	23.3



b)

Table 4. Start and finish temperatures of ferritic and pearlitic transformations for S355J2 steel for various cooling rates

Tabela 4. Temperatury początku i końca przemian fazowych ferrytu i perlitu dla stali S355J2 przy różnych szybkościach chłodzenia

Cooling rate	F_{s}	$F_f = P_s$	P_f	ΔP_{s-f}
[°C/s]	[°C]	[°C]	[°C]	[°C]
0.1	787	666	634	32
0.5	773	654	618	36
1	759	646	595	51
2	753	636	586	50
4	743	622	588	34

Fig. 5. Microstructure of steel S355J2 after cooling with rate: a) 0.1°C/s, b) 0.5°C/s, c) 1°C/s, d) 2°C/s, e) 4°C/s Rys. 5. Mikrostruktura stali S355J2 po chłodzeniu z szybkością: a) 0.1°C/s, b) 0.5°C/s, c) 1°C/s, d) 2°C/s, e) 4°C/s

Kinetics of ferritic and pearlitic transformations are shown in Fig. 7. Due to the changes of the cooling rate between the experiments a span of time between start and finish of ferritic and pearlitic transformations becomes larger when the cooling rate decreases (Fig. 7a, c). When normalized time of transformation is used (Fig. 7b, d) one can find that the kinetic of ferritic transformation is almost not affected by the cooling rate whereas the kinetics of pearlitic transformation accelerates and changes with the increasing cooling rate.

Another feature of the quantitative dilatometric analysis model is an excellent possibility to track carbon enrichment of austenite during its decomposition.



Fig. 6. Volume fraction changes with temperature of ferrite (a) and pearlite (b) during austenite decomposition for different cooling rates

Rys. 6. Zmiany udziału objętościowego ferrytu (a) i perlitu (b) w funkcji temperatury podczas rozpadu austenitu dla różnych szybkości chłodzenia



Fig. 7. Kinetics of austenite decomposition into ferrite (a, b) and pearlite (c, d) for different cooling rates Rys. 7. Kinetyki rozpadu austenitu w ferryt (a, b) i perlit (c, d) dla różnych szybkości chłodzenia

The austenite enrichment in carbon in the analyzed experiments is shown in Fig. 8. One can observe that the increasing cooling rate causes that the austenite enrichment takes place in lower temperatures and simultaneously the maximum carbon concentration in austenite (at the end of ferritic transformation) shifts to higher values beyond the extrapolated A_{cm} line. The phenomenon of such a shift raises a question about the criteria of a pearlitic transformation – a concept that a pearlitic transformation starts when the carbon concentration value equals the A_{cm} value at a certain temperature [6] seems to be not applicable in this case.



Fig. 8. Partial Fe-Fe $_3$ C phase diagram of S355J2 steel with curves of average carbon concentration at austenite for different cooling rates

Rys. 8. Częściowy układ równowagi fazowej stali S355J2 z przebiegami średniego stężenia węgla w austenicie dla różnych szybkości chłodzenia

7. CONCLUSIONS

In the present study the quantitative dilatometric analysis model of ferritic and pearlitic transformation during continuous cooling in hypoeutectoid steel has been presented. The model takes into account the differences in the atomic volume of present phases and the carbon enrichment of austenite during ferrite formation. The following conclusions can be made:

- The results of the validation confirm that the model is correctly developed;
- The comparison of the results from the quantitative dilatometric analysis model and quantitative image analysis of the microstructure show an excellent convergence. The fractions of ferrite calculated by the lever rule method are overestimated since this method does not take into account the increasing atomic volume of austenite during ferrite formation;
- The analysis of kinetics of ferritic and pearlitic transformation with normalized transformation time reveals that the kinetic of pearlitic transformation is in greater extent dependent on the cooling rate than the kinetic of ferritic transformation;
- The obtained results of kinetics can be used directly in validation of numerical models for phase transformations;
- The quantitative dilatometric analysis model shows that the increasing cooling rate causes a shift of the maximum carbon concentration in the austenite (at the end of ferritic transformation) beyond the extrapolated A_{cm} line. It raises a question about the criteria of the pearlitic transformation start. This phenomenon is going to be a subject of a further research.

Acknowledgements

The authors are grateful to Dr Władysław Zalecki for providing CCT research results, an introduction to dilatometry and stimulating discussions. The financial support from the statutory research project S0-0907 granted by the Ministry of Science and Higher Education, Republic of Poland is acknowledged.

REFERENCES

- Onink M., Tichelaar F.D., Brakman C.M., Mittemeijer E.J., van der Zwaag S.: Quantitative Analysis of the Dilatation by Decomposition of Fe-C Austenites; Calculation of Volume Change upon Transformation, Zeitschrift für Metallkunde, 1996, 87, 24-32
- Qiu C., van der Zwaag S.: Dilatation measurments of plain carbon steels and their thermodynamic analysis, Steel Research International, 1997, 68, 32-38
- García de Andrés C., Caballero F. G., Capdevila C.: Dilatometric characterization of pearlite dissolution in 0.1C-0.5Mn low carbon low manganese steel, Scripta Materialia, 1998, 38, 1835-1842
- Kop T.A., Sietsma J., van der Zwaag S.: Dilatometric analysis of phase transformations in hypo-eutectoid steels, Journal of Materials Science, 2001, 36, 519-526
- Zhao L., Kop T.A., Rolin V., Sietsma J., Mertens A., Jacques P.J., van der Zwaag S.: Quantitative dilatometric analysis of intercritical annealing in a low-silicon TRIP steel, Journal of Materials Science, 2002, 37, 1585-1591
- Choi S.: Model for estimation of transformation kinetics from the dilatation data during a cooling of hypoeutectoid steels, Materials Science and Engineering A, 2003, 363, 72-80

- 7. Lee S.-J., Lusk M.T., Lee Y.-K.: Conversional model of transformation strain to phase fraction in low alloy steels, Acta Materialia, 2007, 55, 875-882
- San Martín D., Rivera Díaz del Castillo P.E.J., García de Andrés C.: In situ study of austenite formation by dilatometry in a low carbon microalloyed steel, Scripta Materialia, 2008, 58, 926-929
- Onink M., Brakman C.M., Tichelaar F.D., Mittemeijer E.J., van der Zwaag S.: The Lattice Parameters of Austenite and Ferrite in Fe-C Alloys as Functions of Carbon Concentration and Temperature, Scripta Metallurgica et Materialia, 1993, 29, 1011-1016
- Deufhard P.: Newton Methods for Nonlinear Problems. Affine Invariance and Adaptive Algorithms, Springer Series in Computational Mathematics, 35, Berlin, Springer, 2004
 Andersson J.O., Helander T., Höglund L., Shi P.F., and Sund-
- Andersson J.O., Helander T., Höglund L., Shi P.F., and Sundman B.: Thermo-Calc and DICTRA, Computational tools for materials science, Calphad, 2002, 26, 273-312.
- Szala J., MetIlo v12.1 instruction manual, Katowice, 2009, unpublished