A R C H I V E S O F M E T A L L U R G Y A N D M A T E R I A L S
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B. PAWŁOWSKI*

DETERMINATION OF CRITICAL POINTS OF HYPOEUTECTOID STEELS

WYZNACZANIE PUNKTÓW KRYTYCZNYCH W STALACH PODEUTEKTOIDALNYCH

The paper presents a new approach to interpretation of hypoeutectoid steel dilatograms upon heating between the austenite start and austenite finish temperatures. The proposed derivative curve separation method yields data which accords with the experimental results. Besides the new method appears a useful tool whereby it becomes possible to confirm the evidence reported earlier in literature that when a hypoeutectoid steel heats up to between the A_1 and the A_3 both ferrite and pearlite start to transform into austenite almost at the same time.

Keywords: phase transformation, pearlite dissolution finish temperature, dilatometry, multi-peak fitting

W pracy przedstawiono nowy sposób interpretacji dylatogramów nagrzewania stali podeutektoidalnych w zakresie przemiany perlitu i ferrytu w austenit. Zastosowanie proponowanej metody analizy dylatogramów nagrzewania stali podeutektoidalnych pozwoliło również na potwierdzenie publikowanych wcześniej w literaturze doniesień o jednoczesnym zachodzeniu przemiany perlitu w austenit z przemianą ferrytu w austenit w zakresie temperatury pomiędzy A_1 i A_3.

1. Introduction

The most important heat treatment parameters are the annealing temperature, soaking time and cooling rate [1]. For a given steel the critical points (transformation temperatures) may vary depending on its chemical composition and thermal history, and therefore they must be determined experimentally [2]. As phase transformations occurring in steels are accompanied by expansion or shrinkage [2], the most accurate means whereby the characteristic temperatures of austenite formation during continuous heating can be determined is dilatometry. According to Mehta and Oakwood [3], dilatometry can also be used to explain the reasons for distortions and to predict residual stresses because they allow to measure thermal strains.

Figure 1 presents a graphical method used to determine critical points during heating of a hypoeutectoid, eutectoid and hypereutectoid steel according to the Polish Standard PN 68/H-04500.

Modern dilatometers are fitted with computerised systems which collect dimensional change signals versus temperature to plot a dilatometric curve and also to calculate and plot the derivative of the relative dimensional change with respect to temperature. Figure 2 shows such curves recorded by the Adamel Lhomargy DT1000 dilatometer during heating of a plain-carbon C35 structural steel with an initial ferrite-pearlite microstructure [4]. The pearlite-austenite transformation start temperature (Ac_{1s} in Figure 2) is chosen as the temperature at which the derivative curve first starts to deflect from linearity. The pearlite-austenite transformation finish temperature Ac_{1f} is chosen at the point where the sloping-up part of the derivative curve starts to bend to the right before it returns to another plateau, which moment is chosen as the ferrite to austenite transformation finish temperature Ac_3.

* AGH UNIVERSITY OF SCIENCE AND TECHNOLOGY, FACULTY OF METALS ENGINEERING AND INDUSTRIAL COMPUTER SCIENCE, 30-059 KRAKÓW, 30 MICKIEWICZA AV., POLAND

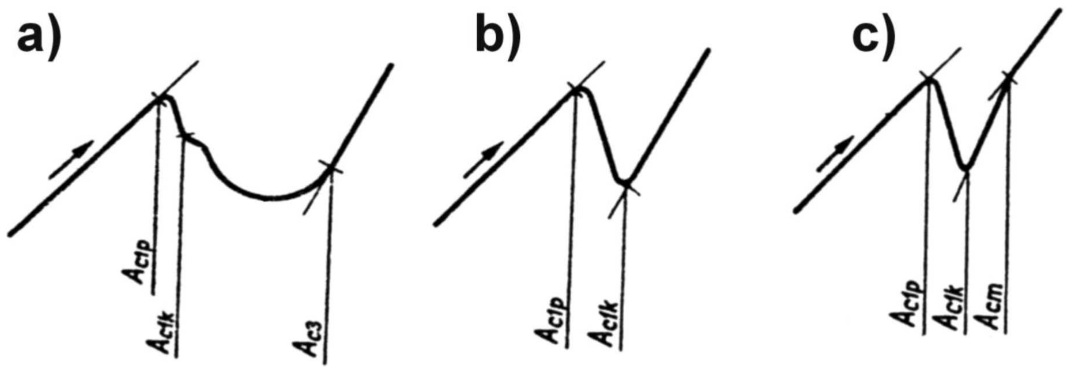

Fig. 1. Graphical determination of critical temperatures in a (a) hypoetectoid, (b) eutectoid and (c) hypereutectoid steel (the subscripts p and k stands for *start* and *finish*, hence the English equivalents for Ac_{1p} and Ac_{1k} are Ac_{1s} and Ac_{1f})

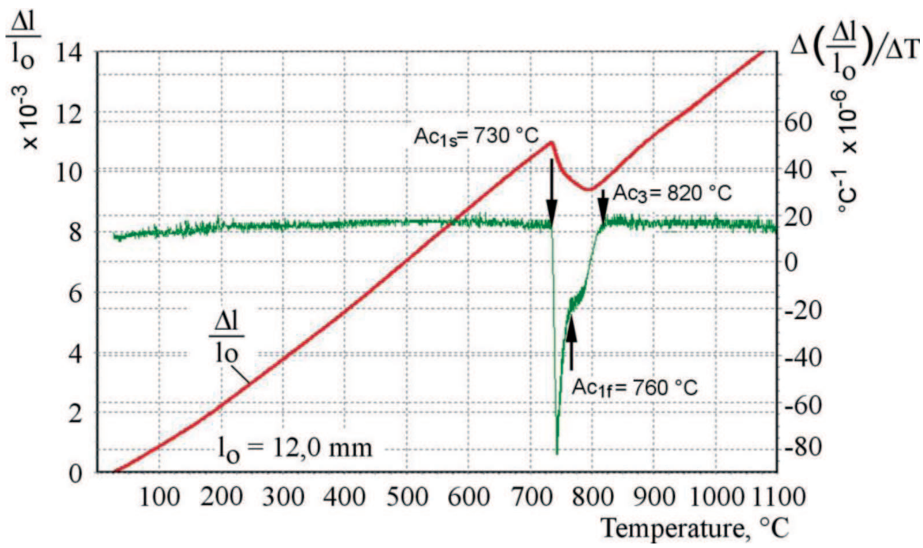

Fig. 2. Relative dimensional change for the C35 hypoeutectoid steel and its derivative vs temperature during heating at 0.05°C/s [4]

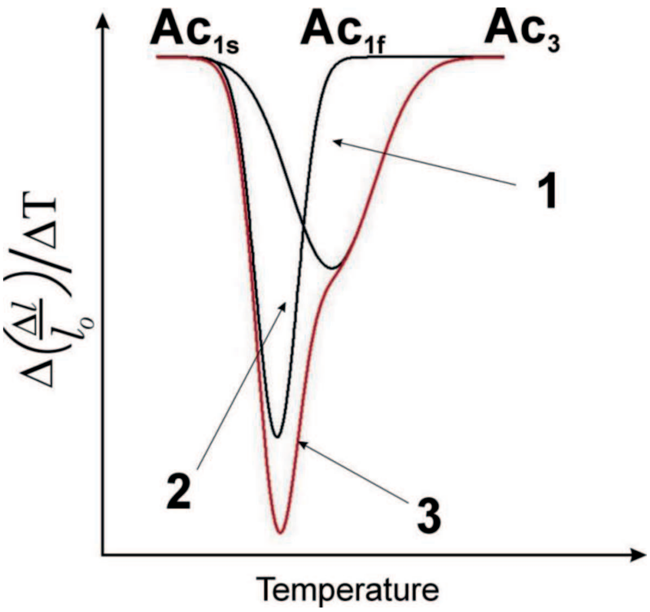

Fig. 3. Derivative curve divided into two component curves corresponding to: 1 – pearlite-austenite transformation, 2 – ferrite-austenite transformation

The main objective of this study was to propose a new approach to the analysis of dilatometric data by numerical treatment of derivative curves.

2. Heating dilatogram and its first derivative

The formation of austenite during heating of initial ferrite-pearlite steel causes volume contraction, observed on dilatometric and derivative curves between the Ac_{1s} and Ac_3 temperature as can be seen in Fig. 2. Thus the derivative curve can be numerically divided into two separate peaks which indicate contraction brought about by the pearlite-austenite and ferrite-austenite transformations (Fig. 3).

The two component curves shown in Fig. 3 can be expressed by Equations (1) and (2), respectively.

$$\Delta(\Delta l/l_0)/\Delta T = 1 - \alpha_P \cdot e^{-\beta_P T^2} \quad (1)$$

$$\Delta(\Delta l/l_0)/\Delta T = 1 - \alpha_F \cdot e^{-\beta_F (T-D)^2} \quad (2)$$

where:

α_P, α_F – coefficients related to the maximum transformation rate

β_P, β_F – coefficients related to the transformation temperature range

D – difference between the pearlite-austenite and ferrite-austenite transformation start temperatures.

It is noteworthy, however, that the derivative curve can be presented as superposition of peaks constructed in a variety of ways depending on the relationship between the maximum rate and temperature range of both transformations.

The algorithm proposed in Fig. 3 requires experimental verification since there is a lot of contradictory information on whether the ferrite-austenite transformation start temperature lies below or above the Ac_{1f}. According to some recent works [5-7] the ferrite-austenite transformation starts above the Ac_{1f} temperature, although Egorov, in his earlier work [8], observed formation of austenite at grain boundaries in ferrite before the Ac_{1f} temperature was reached (Fig. 4). This was later corroborated experimentally by San Martin [9], who found that the nucleation of austenite occurs concurrently both within the pearlite colonies and at grain boundaries in ferrite as well (Fig. 5).

The proposed derivative curve separation method is apparently more accurate then the classical ones presented in Figures 1 and 2. The analysis of experimental data can be easily automated by using either dedicated routines or a commercial curve fitting software. The latter approach was exemplified in Figures 6-8 by means of the OriginLab 8.5 software.

In Figures 6 and 7 the experimental derivative curve from Figure 2 was fitted with two asymmetric double sigmoid peaks represented by Equation (3), whereas in Figure 8 it was fitted with the Voigt peaks represented by Equation (4).

a) b)

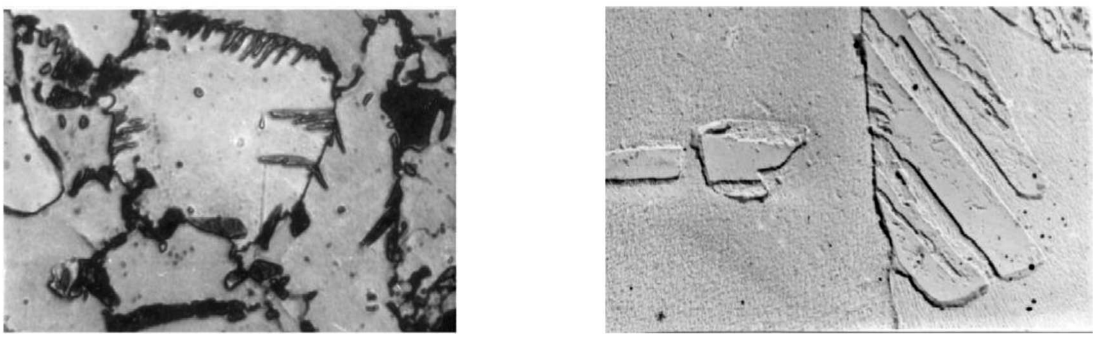

Fig. 4. Nucleation of austenite at ferrite grain boundaries in the 10HSND grade steel: (a) 1000x, (b) 4800x [8]

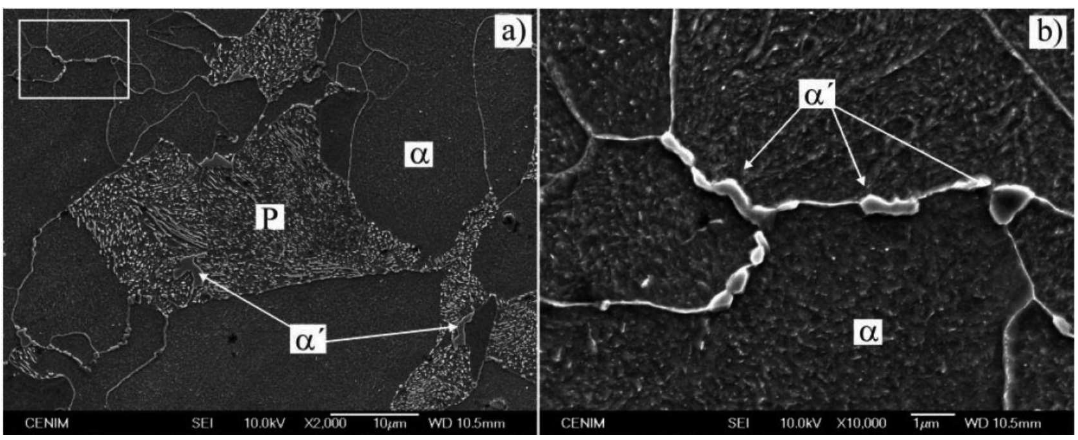

Fig. 5. Nucleation of austenite: (a) inside pearlite colonies and (b) at grain boundaries in ferrite [9]

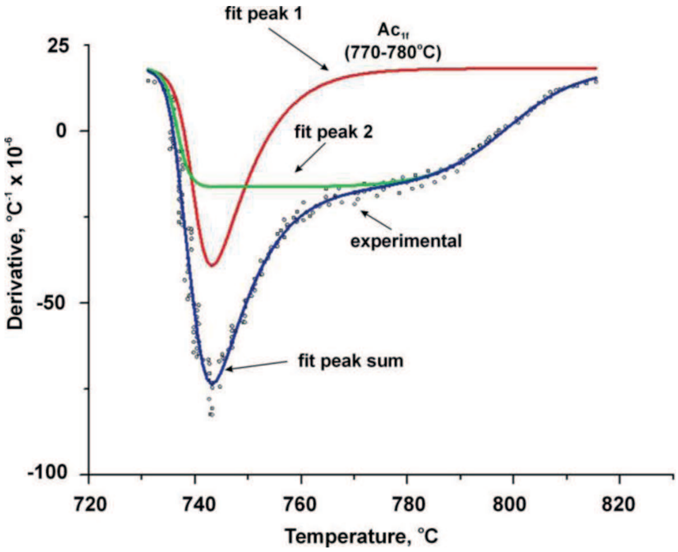

Fig. 6. Derivative peak fitting by superposition of two asymmetric double sigmoid peaks

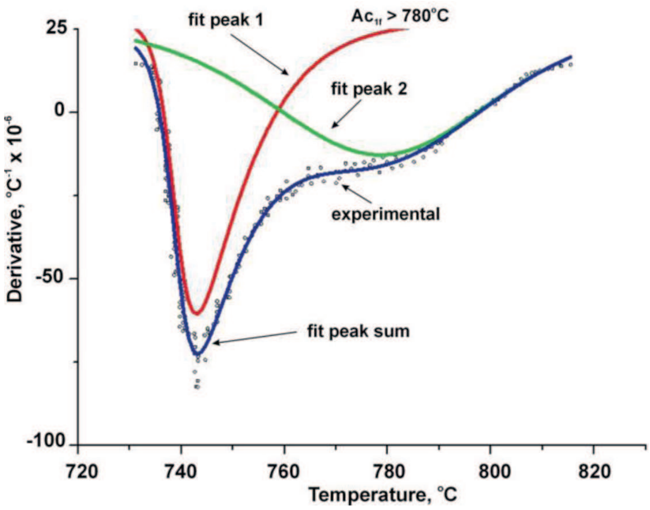

Fig. 7. Another derivative peak fitting by superposition of two asymmetric double sigmoid peaks

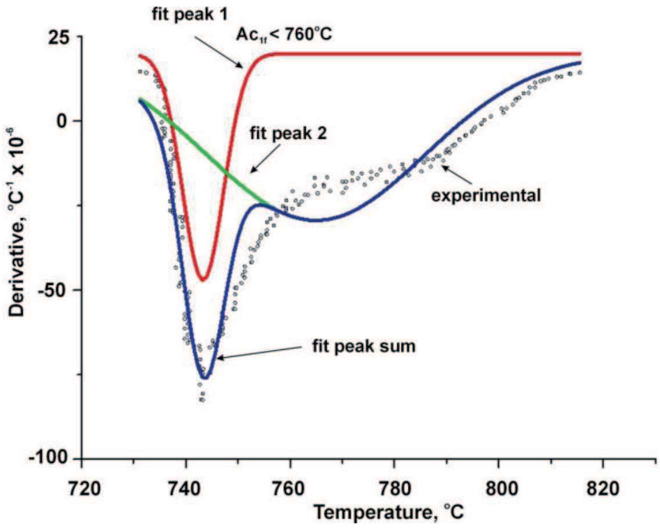

Fig. 8. Derivative peak fitting by superposition of two Voigt peaks

$$y = y_0 + A \frac{1}{1 + e^{-\frac{x - x_c + w_1^{1/2}}{w_2}}} \left(1 - \frac{1}{1 + e^{-\frac{x - x_c - w_1^{1/2}}{w_3}}}\right) \quad (3)$$

$$y = y_0 + A \frac{2 \ln 2 W_L}{\pi^{3/2} W_G^2}$$

$$\int_{-\infty}^{\infty} \frac{e^{-t^2}}{\left(\sqrt{\ln 2}\frac{W_L}{W_G}\right)^2 + \left(\sqrt{4 \ln 2}\frac{x - x_c}{W_G} - t\right)^2} dt \quad (4)$$

The coefficients used in Equations (3) and (4) in order to construct the two component peaks are provided in Tables 1 and 2, respectively.

The coefficients of determination R^2 interpreted as the proportion of observed variation that can be explained by the two nonlinear regression models, i.e. fitting with the asymmetric double sigmoid peaks (Figures 6 and 7) and Voigt peaks (Figure 8) are 0.96 and 0.88, respectively. This clearly indicates that the asymmetric double sigmoid function is almost ideally suited for the peak separation procedure, however, accurate determination of both the ferrite-austenite transformation start temperature and the pearlite-austenite transformation start temperature (Ac_{1s}) using the two fitted peaks requires developing a separate algorithm which will determine the characteristic temperatures basing on an arbitrary criterion how much the points on the two peaks can depart from the baseline established on the prior-to-transformation side of the peak. Therefore additional research is needed which would involve quenching of a hypoeutectoid steel from various temperatures lying above the Ac_{1s} temperature in order to find the onset of transformations metallographically and thus be able to calibrate the calculation routine.

Nevertheless, the data presented in Figures 6 and 7 apparently corroborates the evidence reported in Refs [8,9] that when a hypoeutectoid steel heats up above the A_1 temperature the nucleation of austenite in pearlite concurs with its nucleation in ferrite.

TABLE 1

Asymmetric double sigmoid function coefficients in Equation (3)

offset y_0	**center x_c**	**amplitude A**	**width w_1**	**width w_2**	**width w_3**
Figure 6 peak 1					
18,35907	740.31569	-169.89941	1.81964E-20	1.58565	6.69649
Figure 6 peak 2					
18.35907	767.68465	-34.569	62.18739	1.15475	6.80419
Figure 7 peak 1					
27.48978	739.26372	-249.61504	1.77909E-13	1.85207	9.20431
Figure 7 peak 2					
27.48978	778.89859	-98.17574	16.25835	14.45192	13.88804

TABLE 2

Voigt function coefficients in Equation (4)

offset y_0	**center x_c**	**amplitude A**	**Gaussian full width at half maximum W_G**	**Lorentzian full width at half maximum W_L**
Figure 8 peak 1				
20.05946	743.26694	-663.82574	9.28131	8.12954E-13
Figure 8 peak 2				
20.05946	764.89067	-2587.03491	49.22041	6.13068E-14

3. Conclusions

- The use of the asymmetric double sigmoid function to dilatometric derivative curve fitting yields higher determination coefficient (R^2 = 0.96) as compared to the Voigt function (R^2 = 0.88).
- The contraction indicated by the derivative curve results from two independent phenomena, i.e. from the pearlite-austenite transformation and from the ferrite-austenite transformation.
- The presented peak fitting data provides forensic evidence proving that when a hypoeutectoid steel heats up to between the A_1 and the A_3 the transformation of ferrite into austenite begins concurrently with the eutectoid reaction.

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