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
Volume 59 2014 Issue 3
DOI: 10.2478/amm-2014-0202

B. PAWŁOWSKI*, P. BAŁA*, R. DZIURKA*

IMPROPER INTERPRETATION OF DILATOMETRIC DATA FOR COOLING TRANSFORMATION IN STEELS

NIEPRAWIDŁOWA INTERPRETACJA DYLATOGRAMÓW DLA PRZEMIAN PODCZAS CHŁODZENIA STALI

Dilatometry is the most commonly method of producing CCT diagrams and analyzing phase transformations during cooling (as well as transformations during heating) and it permits the real-time monitoring of the extent of reaction in terms of dimensional changes due to phase transformation. All 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. Unfortunately, elaborated by dilatometers manufacturers software, in many cases could lead to wrong interpretation of phase transformations during cooling.

Keywords: dilatogram, derivative, phase transformations during cooling, software

Dylatometria jest najczęściej stosowana technika badawczą przy tworzeniu wykresów CTPc i analizie przemian fazowych podczas chłodzenia oraz nagrzewania. Umożliwia również śledzenie na bieżąco postępu przemiany dzięki rejestracji zmian wymiarów próbki w trakcie przemiany. Wszystkie współczesne dylatometry są wyposażone w systemy komputerowe sterowania i akwizycji danych pomiarowych umożliwiające obserwację dylatogramu w trakcie chłodzenia czy nagrzewania jak również obliczenie i narysowanie po zakończeniu eksperymentu wykresu różniczki (pierwszej pochodnej) w funkcji temperatury. Niestety, fabryczne oprogramowanie w wielu przypadkach może prowadzić do błędnej interpretacji zjawisk podczas przemian fazowych w trakcie chłodzenia.

1. Introduction

Transformation temperatures for steels are typically determined by Differential Scanning Calorimetry (DSC) or, as phase transformations occurring in steels are accompanied by expansion or shrinkage, by use of dilatometric technique (sometimes by a combination of dilatometry and metallographic methods). 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 and Fig. 1 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 [1]. Critical points – Ac_{1s} (pearlite to austenite transformation start temperature), Ac_{1f} (pearlite to austenite transformation finish temperature) and Ac_3 (ferrite to austenite transformation finish temperature) are indicated by arrows on the derivative curve in Fig. 1.

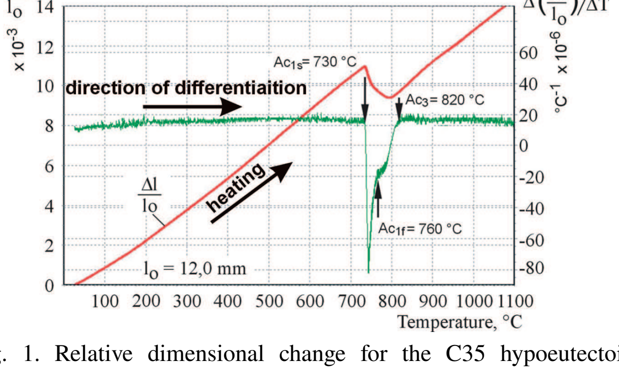

Fig. 1. Relative dimensional change for the C35 hypoeutectoid steel and its derivative vs temperature during heating at 0.05°C/s, Adamel-Lhomargy DT1000 dilatometer [1]

2. Transformation during heating

As can be seen in Fig. 1, direction of differentiation is the same as the direction of temperature changes, i.e. from the lowest to the highest temperature. Thus, contraction on the dilatometric curve, caused by the austenite formation between Ac_{1s} and Ac_3 temperature, is obviously reflected on the derivative. Similar case is illustrated in Fig. 2, presenting heating dilatogram and its first derivative for HS18-0-1 steel heated from as-quenched state at 0.05°C/s [2].

In case when effects of volume change (caused by phase transformation) are clearly shown on the dilatometric curve (as in Fig. 1), transformation temperatures may be determined by the choice of the few different evaluation methods, as it is

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

described for example in paper [3] and presented graphically in Fig. 3.

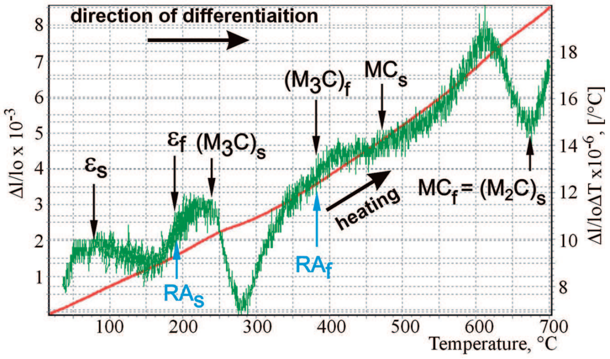

Fig. 2. Dilatogram and its first derivative for HS18-0-1 steel heated from as-quenched state at 0.05°C/s, Adamel-Lhomargy DT1000 dilatometer [2]

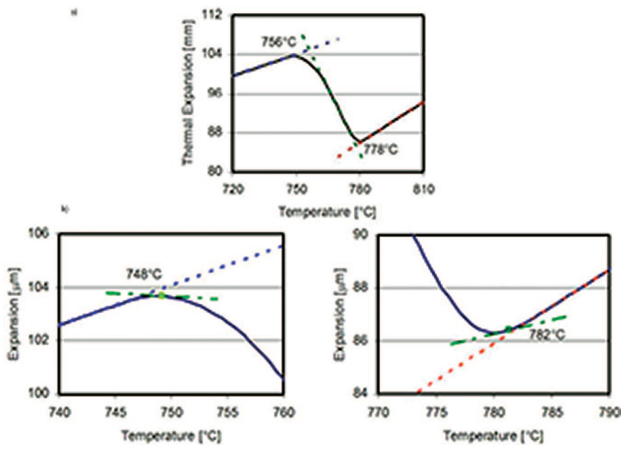

Fig. 3. Determination of transformation temperatures: a) three tangents method, b) transformation temperature as the intersection of linear section and tangent at point of 10° deflection [3]

From Fig. 2 we can see that although the specimen shrinkage or expansion is less pronounced on dilatometric curve such sample volume changes are very well visible on first derivative curve and it is easy to indicate phase transformation start and/or finish temperatures.

Another example of the usefulness of dilatometric first derivative curve for phase transformation analysis during heating was presented in paper [4] where non-isothermal precipitation of copper-rich phase in 17-4 PH steel was investigated (Fig. 4).

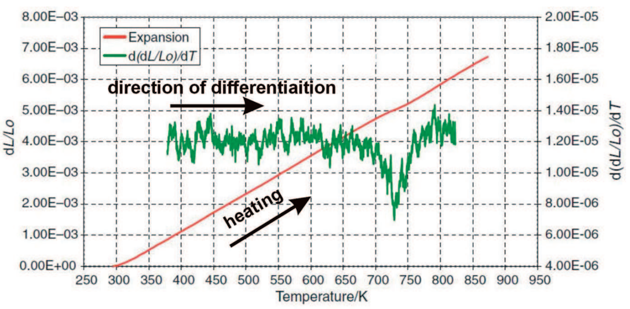

Fig. 4. Expansion curve and its derivative of an as-quenched 17-4 PH steel sample, 2 K min^{-1} heating rate, Netzsch 402 ES dilatometer [4]

3. Transformation during cooling

In the literature there are very few cooling dilatograms with derivatives although such dilatometric examinations are the basis for the development of the continuous cooling transformation diagrams (CCT diagrams). Such dilatograms are presented in Fig. 5 [2] and Fig. 6 [5].

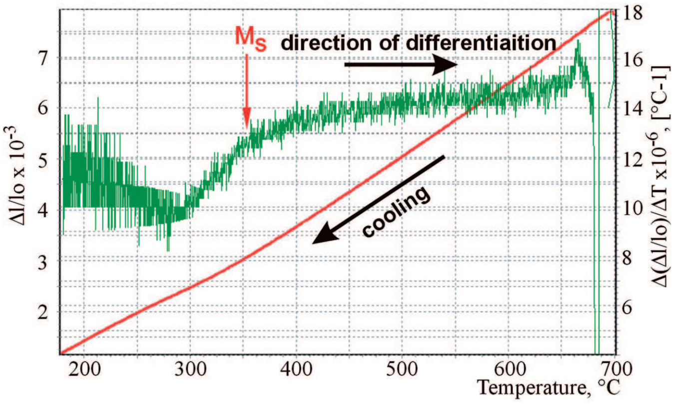

Fig. 5. Cooling dilatogram and its first derivative, Adamel-Lhomargy DT1000 dilatometer [2]

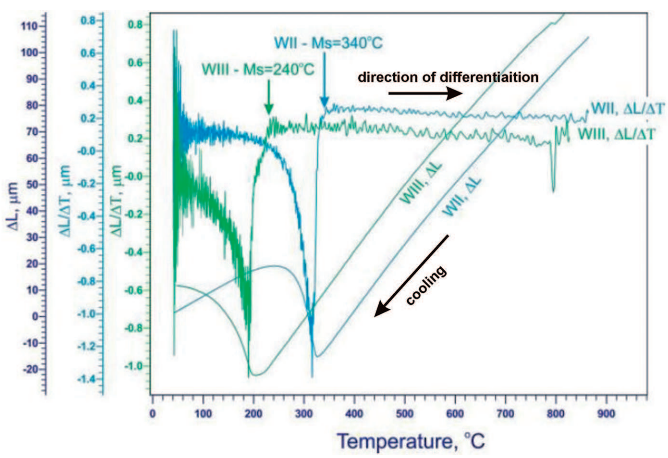

Fig. 6. Cooling dilatogram and its first derivative, RITA L78 dilatometer [5]

As can be seen in Fig. 5 and Fig. 6, direction of differentiation is not the same as the direction of temperature changes, i.e. from the highest to the lowest temperature, thus specimen expansion starting at martensite start temperature Ms which is sufficiently clearly visible on the dilatogram is reflected as shrinkage on the derivative curve. In this case, although the erroneous process of differentiation, it is evident for all that martensitic transformation is always associated with the increase of the volume. Of course, a possible solution to be sure about the direction of the volume change is to load the curve over time and calculate the derivation over time, as it is presented in Fig. 7 [6].

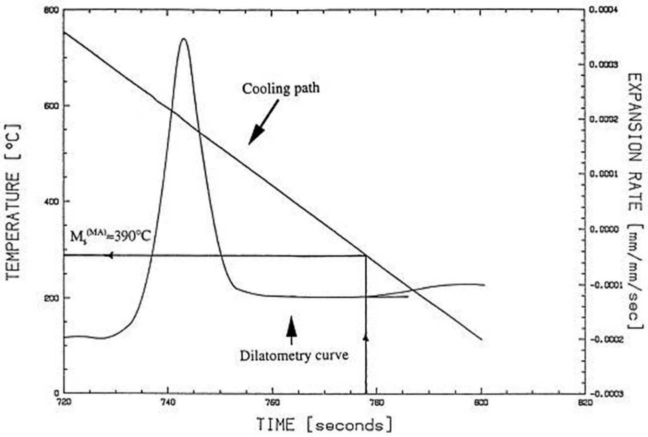

Fig. 7. Thermal and derived dilatation-time analysis indicating the martensite start temperature, Theta dilatometer [6]

Cooling dilatogram presented in Fig. 2 was proper derived for this work and its first derivative is presented in Fig. 8.

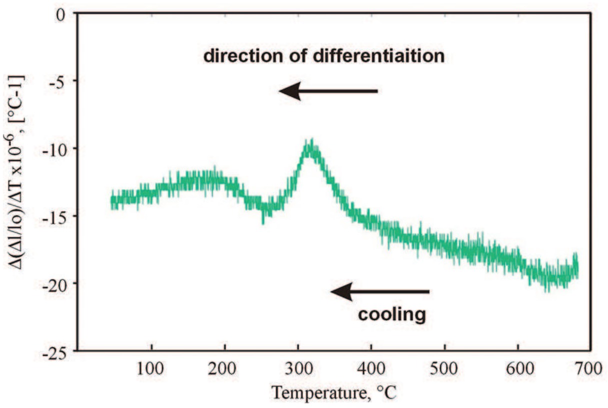

Fig. 8. Proper first derivative of dilatogram presented in Figure 5

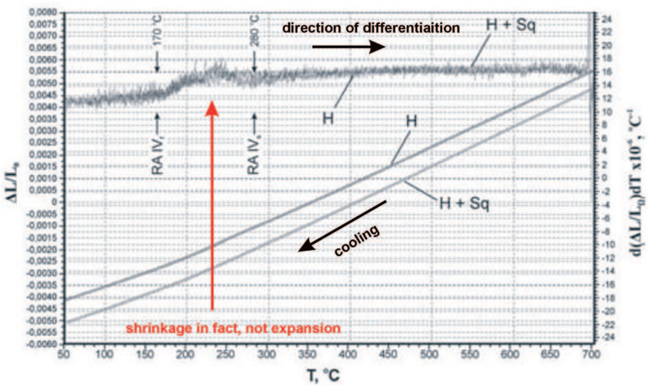

Fig. 9. Dilatograms of cooling with a rate of 0.05°C/s of C110 steel samples previously hardened (H) and hardened and later subquenched (H+Sq), heated to 700°C and the corresponding differential curves, Adamel-Lhomargy DT1000 dilatometer [7]

However, for some cooling dilatograms volume changes are not so clear and then the proper process of differentiation allows to define temperature range and nature of phase transformation like for heating dilatogram shown in Fig. 1. Unfortunately, as it is known to the authors, every factory dilatometers software is supplied only with the option of differentiation over temperature from the lowest to highest temperature, which can lead in some cases to completely erroneous interpretation of experimental results. Such an example can be seen in Fig. 9, where the erroneous process of differentiation has led the author [7] to conclude that there has been retained austenite to martensite transformation (always associated with the expansion) during cooling in the temperature range 280-170°C [7].

4. Conclusions

On the basis of the presented above information it can be said that:

- Dilatometers software is supplied only with the option of differentiation over temperature from the lowest to highest temperature regardless of whether the experiment is heating or cooling.
- Such improper differentiation manner can lead in some cases of cooling investigations to completely erroneous interpretation of experimental results.
- A possible solution to be sure about the direction of the volume change is to load the curve over time and calculate the derivation over time.

REFERENCES

[1] B. Pawłowski, Determination of critical points of hypoeutectoid steels, Archives of Metallurgy and Materials **49**, 2, 957-962 (2012).

[2] P. Bała, J. Krawczyk, Transformations during quenching and tempering of hot-work tool steel, METAL 2009, 18th International Conference on Metallurgy and Materials, Hradec nad Moravici, Czech Republic, 64-71, May 19-21 2009.

[3] P. Motyčka, M. Kōvėr, Evaluation methods of dilatometer curves of phase transformations, COMAT 2012, 2nd International Conference on Recent Trends in Structural Materials, November 21st-22nd 2012, Parkhotel Plzen, Czech Republic.

[4] B. Rivolta, R. Gerosa, On the non-isothermal precipitation of copper-rich phase in 17-4 PH stainless steel using dilatometric technique. Journal of Thermal Analysis and Calorimetry **102**, 857-862 (2010).

[5] R. Dziurka, J. Pacyna, T. Tokarski, Effect of heating rate on phase transformations during tempering of Cr-Mn-Mo alloy steels, METAL 2013, 22nd International Conference on Metallurgy and Materials, Brno, Czech Republic, 700-705, May 15-17 2013.

[6] S.S. Ghasemi Banadkouki, D.P. Dunne, Formation of ferritic products during continuous cooling of Cu-bearing HSLA steel, ISIJ International **46**, 5, 759-768 (2006).

[7] J. Pacyna, Dilatometric investigations of phase transformations at heating and cooling of hardened, unalloyed, high carbon steels, Journal of Achievements in Materials and Manufacturing Engineering **46**, 1, 7-17 (2011).

Received: 20 March 2014.