Techniques for Simplifying the Construction of a Reference Temperature for Single Sensor Differential Thermal Analysis (SS-DTA)

Wutipong Nieampradit, Sarawan Wongsa, and Isaratat Phung-on

***Abstract*—**SS-DTA is an efficient tool for analysing phase transformations of metals during actual thermal processing conditions. SS-DTA involves the implementation of the differential temperature profile between the measured temperature and the reference temperature for detecting any phase changes of the investigated material. In this paper, we applied and compared several data modelling techniques to obtain the reference curve for SS-DTA technique. These models include the polynomial model, the piecewise polynomial model, and the piecewise linear model. Tests on a stabilised grade stainless steel SUS 321 were constructed to evaluate the performances of the proposed techniques in comparison with the conventional reference curve estimation approach.

***Keywords*—**Nonlinear approximation, Phase transformation, Reference thermal cycle, Thermal analysis

I. Introduction

THEquality of a weldment can be controlled by controlling its microstructure. However, this could be done only after welding is completed and a typical microstructure analysis must be performed in which it could damage the sample. To avoid these limitations, a non-invasive thermal analysis method that can in-situ determine phase transformations of the investigated specimen called the single sensor differential thermal analysis (SS-DTA) has been developed and applied in weld metal and other thermal processing applications[1-4].

SS-DTA is a technique to investigate phase transformation properties and structural changes of material by comparing the temperature recorded in a measured specimen against a reference thermal history. Similar to the differential thermal analysis (DTA), when the investigated specimen undergoes any phase changes during the analysis, the phase transformations impact the heating and cooling rates of the material. The occurrence of phase transformations can be revealed by plotting the difference in the tested specimen against that of the reference one over time or the current temperature. However, in contrast to the traditional DTA, SS-DTA needs neither a reference temperature sensor nor a reference specimen, thus simplifying the measurement and allowing its application in actual thermal processing conditions [4].

In a typical SS-DTA study, the construction of reference temperature profile is usually obtained by optimising the parameters in the analytical formulae or by a modelling of the heat flow in the tested sample [1-4]. The adequate closeness between the estimated reference curve and the measured data plays a significant role in phase transformation accuracy and sensitivity of SS-DTA [4]. The present work proposes and compares the utility of three data modelling techniques to simplify the conventional SS-DTA which is based on analytical formulae.

This paper is organised as follows. In Section II, we provide a general framework of SS-DTA technique, including the conventional approach to generate the reference temperature, and develop data modelling techniques for construction of the reference thermal profile. In Section III, we present the results for the experiment data obtained from a welded stainless steel. The last section provides relevant discussion of models and their potential applications.

II. Methods

SS-DTA is based on the calculation of temperature deviation (ΔT) or *delta T* for each time instant *t* between the measured temperature of the investigated specimen and the reference temperature (T_r) as shown in (1)

$$\Delta T(t) = T(t) - T_r(t) \tag{1}$$

A sudden change of the rate at which ΔT decreases as a function of T indicates phase transformations of the sample. The reference temperature is usually calculated using numerical modelling of the thermal cycle equations in (2) and (3) [4]:

$$T = T_0 + \theta_k \left(\frac{\Delta t}{t}\right)^{1/k} \cdot \exp\left(-\frac{\theta_k^k \Delta t}{ket(T_p - T_0)^k}\right) \tag{2}$$

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$$\frac{1}{\theta_k} = \left(\frac{1}{(500 - T_0)^k} - \frac{1}{(800 - T_0)^k} \right) \quad (3)$$

where T_0 is the initial temperature, T_P is the maximum temperature, Δt is the cooling time between eight hundred and five hundred degrees Celsius, θ is an expression used for simplifying the way (2) is presented, and k is related to heat extraction capacity of the processed sample.

If the values of physical parameters in (2) and (3) are known, then we can generate a reference curve and the differential thermal profile in (1) can be obtained accordingly. The data inflection points of this curve are used as reference points to determine the phase transformations starting and finishing points.

The sensitivity in determining the transformation heat effects depends on how close to the measured temperature the reference curve is [4]. Therefore, the problem of reference curve construction is transformed to a search for a set of optimal parameters that gives the closest reference thermal profile to the measured thermal history. This is usually solved by using available optimisation procedures. Alternatively, the reference thermal cycle in (2) can be approximated by nonlinear approximation techniques.

A. Polynomial Approximation

A common choice for generating a reference thermal cycle in thermal analysis is a polynomial function [5]. Polynomials are usually chosen because there are efficient methods both for determining and for evaluating them. The polynomial function-based reference curve is in the following form:

$$T_r^P(t) = a_n t^n, a_{n-1} t^{n-1} + \ldots + a_1 t + a_0 \quad (4)$$

where T_r^P is the reference curve approximated as a polynomial function, n is the degree or the order of the polynomial, and a_0, a_1, …, a_n are the coefficients of the polynomial.

The polynomial degree determines the number of turning points where the polynomial function changes direction from increasing to decreasing, or vice versa. The higher degrees of polynomial usually result in more accuracy of curve approximation. However, when the order is greater than an order, the accuracy has small effects with the order. On the other hand, when the order is high, more calculation time is required.

B. 4 –Segment Piecewise Nonlinear Approximation

The basic ideal of piecewise nonlinear approximation is that we represent the reference thermal profile by a different polynomial function on each interval between data points. Piecewise polynomial (spline) approximation pieces together local polynomial approximants. The method is considered because it may capture local nonlinear behaviours of the reference curve better than using a single polynomial function which captures only the global nonlinearity of reference curve.

The polynomial degree and the number of segments determine the computation complexity of this approximation method. Here, we limit the number of segments to be four with equal segment width and let the polynomial degree control the ability of complexity approximation. The 4-segment piecewise nonlinear approximation can be written as

$$T_r^{PWNL}(t) = \sum_{i=1}^{4} P_i(t) \quad (5)$$

$$P_i(t) = \begin{cases} a_{n,i} t^n + a_{n-1,i} t^{n-1} + \ldots + a_{1,i} t + a_{0,i}, (i-1)W_p \le t \le iW_p \\ 0, \text{otherwise} \end{cases} \quad (6)$$

where $P_i(t)$ is the local polynomial function of data segment $i, a_{n,i}, a_{n-1,i}, \ldots, a_{0,i}$ are the coefficients of local polynomial $P_i(t)$ and W_p is the segment width in time unit which is equal to the total number of data points divided by four, assuming that the data are uniformly sampled.

C. Piecewise Linear Approximation

The last method approximates the nonlinear reference temperature by a piecewise linear one. The segments can have different widths or equal width; the former known as a non-uniform segmentation and the latter known as a uniform segmentation.

The piecewise linear approximation can be formulated as a nonlinear optimisation problem to find optimal slopes, offsets, and breakpoints of the local linear functions [6]. To simplify the piecewise linear approximation problem, here we consider the uniform segmentation case, where the piecewise linear function is represented as

$$T_r^{PWL}(t) = \sum_{i=1}^{M} f_i(t) \quad (7)$$

$$f_i(t) = \begin{cases} m_i t + b_i, l_i \le t \le u_i \\ 0, \text{otherwise} \end{cases} \quad (8)$$

Where M is the number of segments over the ranges of considered time, m_i and b_i are the constant coefficients of segment i, $l_i = (i\text{-}1) \cdot W$ and $u_i = i \cdot W$ with W being the segment width in time unit.

As the piecewise linear function in (8) can be realised as a straight line between two adjacent data points and no optimization routine is needed, the complexity of nonlinear approximation is greatly reduced. We can use a large number of segments to increase the accuracy of approximation.

III. Results

In this section, we make some experiments to demonstrate the validation of the nonlinear approximation techniques. The experiment was implemented Gas Tungsten Arc Welding (GTAW) on stabilized grade stainless steel SUS 321. The specimens of sizes 81 x 63 x 6mm were prepared. A set of thermocouples were attached to the specimen at the area adjacent to the prospective fusion boundary of the weldment.

The sampling rate used for collecting temperature profile was 2 kHz recorded to a computer. The signals from thermocouples were filtered by a moving average filter to reduce the noise and make the heat effects of phase transformations more clearly observable [4].

After obtaining the measurement of temperature profile of specimen, we applied the SS-DTA technique by using reference curve approximation techniques presented in the previous section. For the analytical formula-based technique, we approximated T_o, T_P, and Δt from the measured data as 35 °C, 918.42 °C, and 32.63 s, respectively. As suggested in [4], the optimal values of parameters Δt and k can be solved by an optimisation technique. The optimisation procedure implemented in the present study makes use of the nonlinear least squares routine *lsqnonlin* of MATLAB with Levenberg-Marguardt (LMA) algorithm. The bounds of Δt and k are [25, 40] s and [1, 2], respectively. Note that the bounds of k are selected based on the thermal cycle equations of the welding of thick plates and thin plates [7]. The optimal values of Δt and k obtained are 30.7175s and 1.9985, respectively.

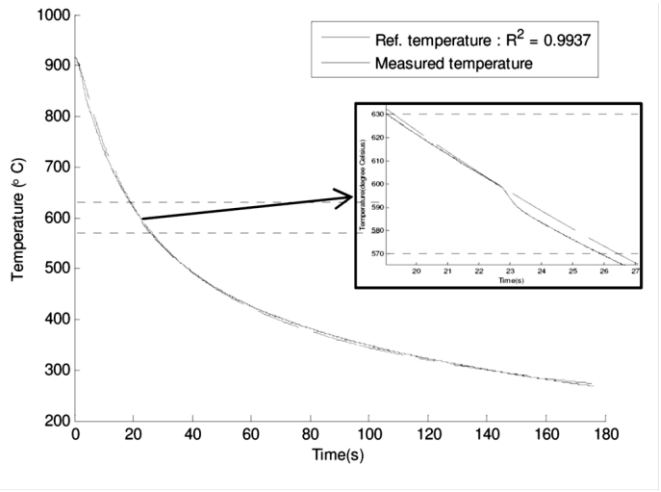
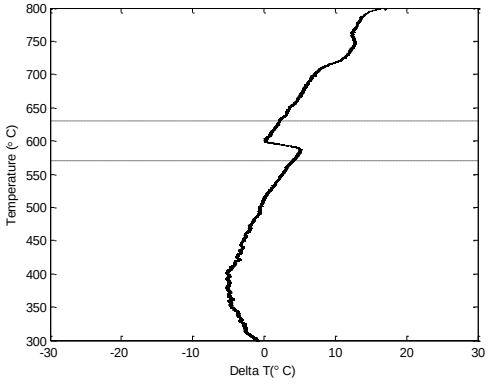
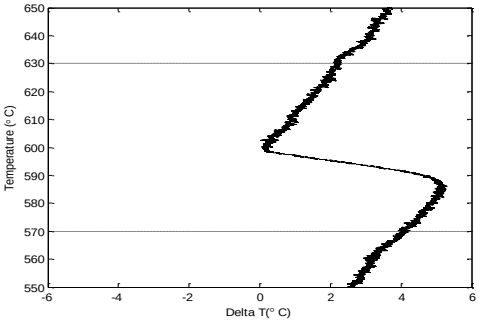


Fig. 1 Cooling temperature profiles between the measured temperature and reference temperature of thermal cycle equations and their zoomed-in view.

The results of analytical-formula-based SS-DTA are shown in Figs.1 and 2. The R^2 is used as a measure of goodness-of-fit of the reference curve to the measured data.



(a)



(b)

Fig. 2 SS-DTA of the thermal cycle equation (a) and its zoomed-in view (b). The horizontal dash lines indicate the start and finish transformation temperatures

We can see that there are significant changes of ΔT at around 570°C – 630°C. These changes appear as local deviation over the $T(\Delta T)$ curve in Fig. 2, whose beginnings and ends coincide with the start and finish of the phase transformation. Note that the temperature from 570°C – 630°C is where Chromium Carbide ($Cr_{23}C_6$) precipitates can be formed. In order to confirm the precipitates, the specimen was analysed by optical microscope, as shown in Fig. 3. It can be clearly seen that there are $Cr_{23}C_6$ precipitates along the grain boundary.

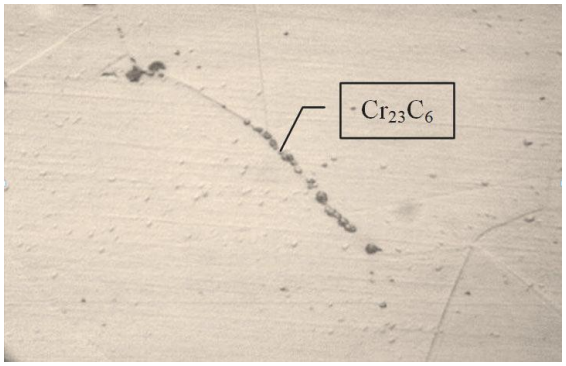


Fig. 3 Microstructure of SUS 321 of $Cr_{23}C_6$

We then applied the other three considered approximation methods to investigate their performance in phase transformation detection. For polynomial approximation, we started from the cubic function, as suggested in [5], and increased the polynomial degree until there was no significant improvement of R^2. The results of the comparison, presented in Figs. 4 and 5, show that the cubic function is not sufficient to capture the phase transformations, whereas the other higher degrees provide quite similar observations during the start and finish phase transformation temperatures with higher R^2, when compared with the thermal cycle equations.

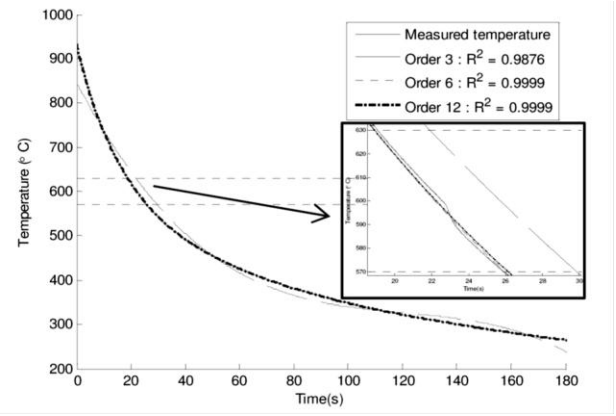


Fig. 4 Cooling temperature profiles estimated by polynomials degrees 3, 6, and 12 and their zoomed-in view.

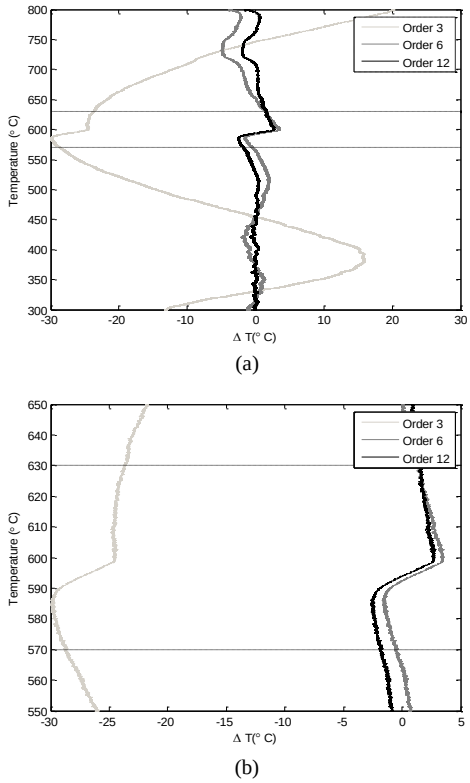


Fig. 5 SS-DTA results obtained from (a) polynomials degrees 3, 6, and 12 and (b) the zoomed-in view. The horizontal dash lines indicate the start and finish transformation temperatures

The next method is the 4-segment piecewise nonlinear approximation. The results are shown in Figs.6 and 7 for local polynomials at the orders of 3, 6, and 12, respectively. As expected, the 4-segment piecewise polynomial can approximate the nonlinear cooling temperature better than the single polynomial function with the same polynomial degree. In other words, instead of approximating the reference temperature by a single polynomial of high degree, one can approximate it by piecewise polynomials of much lower degree to obtain the same accuracy.

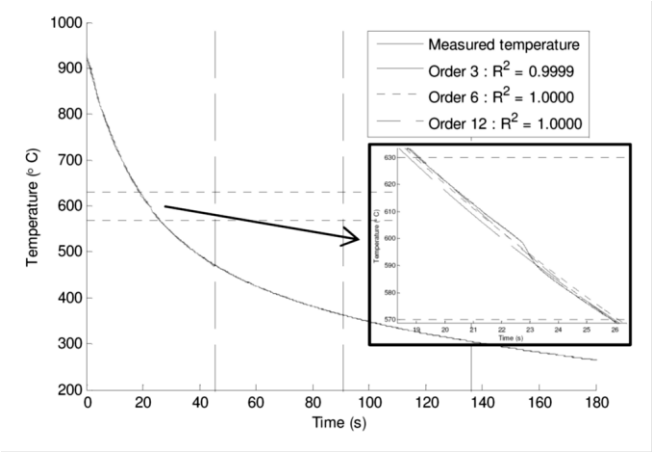


Fig. 6 Cooling temperature profile of curve fitting from 4-segment piecewise polynomials at the orders of 3, 6, and 10

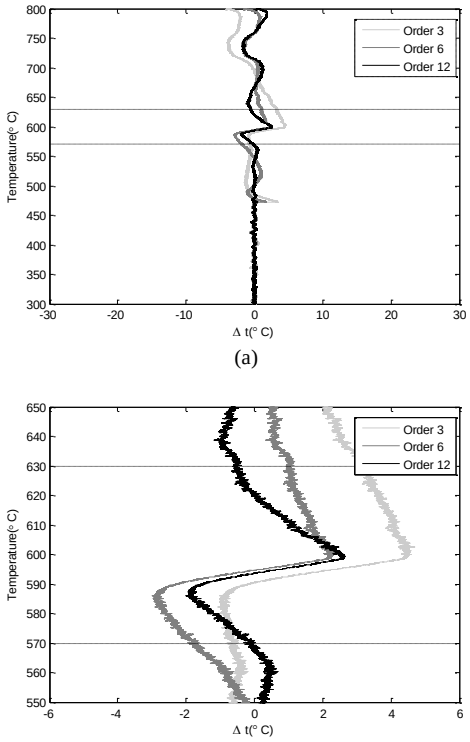


Fig. 7 SS-DTA results obtained from (a) the 4-segment piecewise polynomials at the orders of 3, 6, and 12and (b) the zoomed-in view. The horizontal dash lines indicate the start and finish transformation temperatures.

The last method is to use the piecewise linear approximation for constructing the reference cooling curve. The uniform segment widths 0.5 s, 1.5 s, and 3.5 s were tested and their results are shown in Figs. 8 and 9. All the tested widths can indicate the release and absorption of heat. However, with 0.5 s the curve is too fitted to the measured data, leading to smallest ΔT during the transformation period. Even though the 3.5s-segment width could amplify ΔT when the phase changes occur, it also provides a large approximation error, when the phase changes are not present (see Fig. 9). Hence, the 1.5s-segment width seems to be a

reasonable choice for this data set.

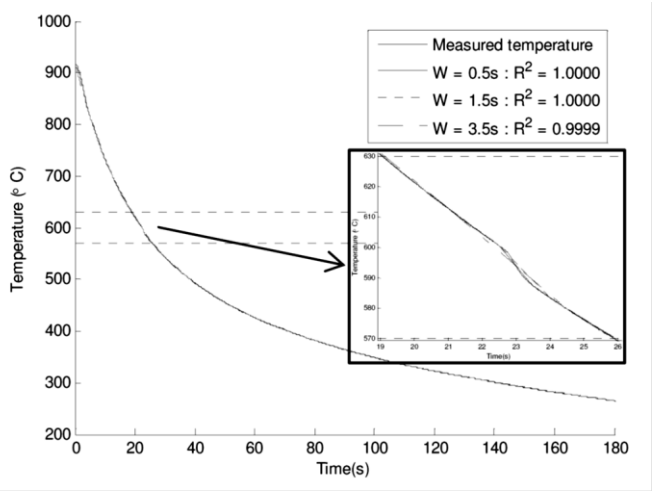
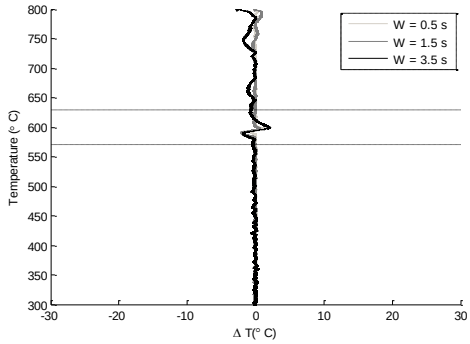
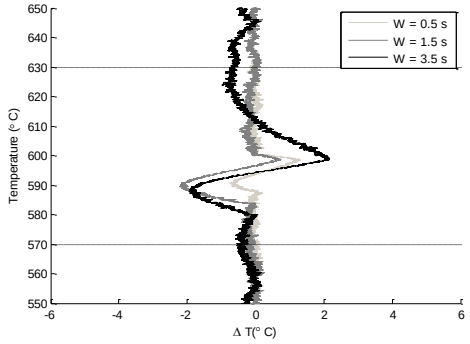


Fig. 8 Cooling temperature profiles of curve fitting by piecewise linear approximation with segment widths 0.5, 1.5, and 3.5 s



(a)



(b)

Fig. 9 SS-DTA results of piecewise linear approximation (a) and their zoomed-in view (b).

IV. Discussion

The objective of this study was to investigate and compare other data modelling techniques that could be used to generate the reference temperature for SS-DTA applications. Remind that what we expect from the SS-DTA technique is its ability to detect the presence of phase transformations of the tested specimen. Therefore, not only the analysis should be able to provide a large ΔT over the phase transformation temperatures, but also it should be able to distinguish between the changes of cooling rates due to the phase transformations and those that are due to the approximation uncertainties, i.e. the measurement noise and approximation error. The latter quality is very important because with high robustness, we do not need a much experienced expert to interpret the results of SS-DTA, therefore simplifying the implementation of an automatic SS-DTA technique.

Consequently, we have invented a quantitative measure of robustness and the ability of SS-DTA techniques to distinguish the changes due to phase transformations from approximation uncertainties, called the *distinguishability index (DI)*. DI is the higher-the-better performance characteristic which is calculated as the ratio between the range of ΔT over the phase transformation temperatures and the range of ΔT over the other temperatures, where the range is the maximum minus the minimum. Table 1 shows the DIs obtained from all considered SS-DTA techniques. The maximum DIs obtained from each SS-DTA technique are highlighted in the table. Obviously, all nonlinear approximation techniques could give a better DI than the thermal cycle equation if the relevant parameters are carefully selected. Moreover, among all the nonlinear approximation techniques the piecewise linear with $W = 0.5$ s gives the best performance in terms of distinguishability.

TABLE I
DISTINGUISABILITY INDEX OF ALL SS-DTA TECHNIQUES

Method	DI
Thermal cycle equation	0.2345
Polynomial degree 3	0.1289
Polynomial degree 6	0.7291
Polynomial degree 12	**1.4579**
4-segment degree 3	0.7631
4-segment degree 6	**1.4835**
4-segment degree 12	1.2401
Piecewise linear (W = 0.5s)	**2.5099**
Piecewise linear (W = 1.5s)	1.7016
Piecewise linear (W = 3.5s)	1.2230

Although we have already applied the piecewise linear approximation of reference temperature and tested on SS-DTA of several metals, such as alloy 617 [8], stainless steel SUS321 [9], and zinc alloy [10] with satisfactory success; however the exploration of other data modelling techniques and the comparison with the traditional SS-DTA have never been made in detail. The results from this study therefore have confirmed the applicability of this simple approximation technique in the proposed applications.

Our future work will consider, for example, the optimal selection of window width of the piecewise linear model and the development of an automatic determination of phase transformations based on our data modelling technique.

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