

# A temperature correction procedure for temperature inhomogeneity in dilatometer specimens

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## Abstract

Dilatometry is a thermo-analytical technique used to measure the expansion or shrinkage of materials during heating or cooling, whether or not in association with a phase transformation. A temperature correction procedure has been developed to correct for the temperature inhomogeneity that exists in an inductively heated specimen during the heating/cooling process and to represent the dilation as a function of a homogeneous temperature. As an example, taking an Fe–5.91 at.% Ni specimen and subjecting it to two different cooling rates, the temperature correction has been performed for the temperature range where the austenite to ferrite phase transformation takes place as well as for the pure austenite and ferrite phases close to the temperature range of the transformation.

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## 1. Introduction

Dilatometry is a technique used to precisely measure the thermal dilation as well as the dilation due to a phase transformation associated with a change of specific volume of the material. It is important to determine precisely both the dilation due to the transformation and the corresponding temperature during the heating/cooling, in order to analyse accurately the phase transformation kinetics.

Relative dilatometers, in which the length change of a specimen is measured relative to the length change of another, reference material, e.g. push-rod dilatometers, are widely used. For small heating and cooling rates (up to  $20 \text{ K min}^{-1}$ ) a resistance heated furnace can provide the desired change of specimen temperature [1]. To achieve high heating and cooling rates a metallic specimen can be heated directly by inductive heating. With this type of heating the dilatation due to phase transformation can also be measured under applied uniaxial compressive or tensile load. A schematic diagram of such a dilatometer is shown in Fig. 1. The temperature of the specimen is con-

trolled by spot welded thermocouples on the specimen. A dilatometer specimen undergoing heat treatment cycles in an inductively heated/cooled environment experiences a temperature inhomogeneity (temperature gradient) in the longitudinal direction [2,3] due to heat loss through the pushrods holding the specimen at its ends in the longitudinal direction. The resulting temperature gradient depends upon the position of the specimen inside the induction coil (the specimen during heat treatment should be positioned in the centre of the induction coil), the quality of spot welding of the thermocouples and the applied heating/cooling rates. Furthermore, the temperature gradient may change during phase transformation due to heat release (recalcescence) which depends on the rate of transformation.

The current practice is to represent the dilation for inductively heated specimens as function of the temperature measured by the thermocouple which controls the applied temperature program. However, such information is biased because the temperature in the specimen is inhomogeneous and the recorded dilation signal cannot be interpreted straightforwardly. In this work an original temperature correction procedure has been developed which allows to extract the specimen dilation as function of a homogeneous temperature. In a separate paper a procedure will be presented for calibration of temperature and length change

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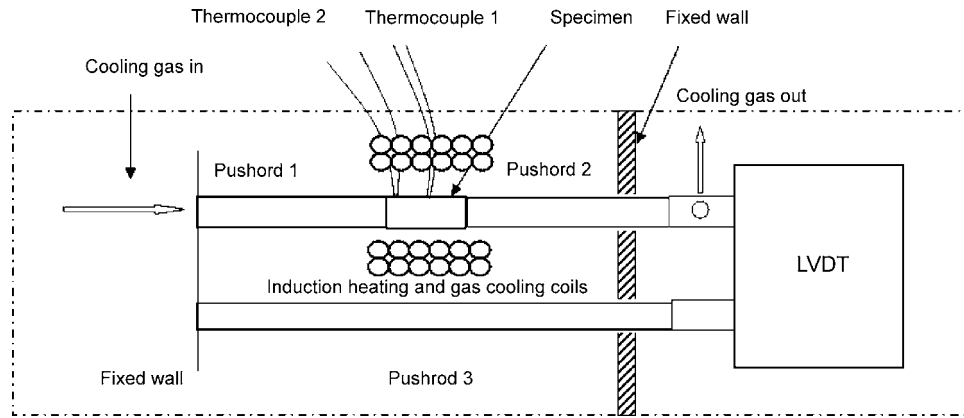


Fig. 1. Schematic diagram showing the spot welded thermocouples on the metallic specimen. Thermocouple 1 controls the imposed heat treatment cycle and thermocouple 2 measures the temperature at the end of the specimen.

upon heating and cooling as well as under uniaxial compressive and tensile loading [4].

## 2. Experimental

### 2.1. Specimen preparation

Bulk high purity Fe (99.98 wt.%) and Ni (99.99 wt.%) were used for the preparation of the alloy. The purity of both Fe and Ni has been indicated by the composition data (determined by inductive coupled plasma-optical emission spectrometry (ICP-OES)) in Table 1. The melting process was carried out in a vacuum-melting furnace, and the molten alloy was cast in a copper mould. The as-cast ingots of 7 mm in diameter were hammered down to rods of 6 mm diameter. In order to achieve a homogeneous distribution of the alloying element all rods were sealed in a quartz container filled with argon gas at  $3 \times 10^4$  Pa, annealed at 1423 K for 100 h and cooled down to the ambient temperature within the furnace. The compositions of the rods were determined by inductive coupled plasma-optical emission spectrometry (ICP-OES). The composition of the alloy was found to be Fe–5.91 at.% Ni. The rods were machined into cylindrical shaped, solid specimens with a length of 10 mm and a diameter of 5 mm.

### 2.2. Dilatometry

A dilatometer DIL-805 A/D (Baehr-Thermoanalysis GmbH) (see Fig. 1), employing inductive heating/cooling was used to measure the thermal dilation of Fe–5.91 at.% Ni specimens upon cooling from the austenite-phase field. The experiments were performed under vacuum ( $6 \times 10^{-6}$  mbar) to avoid oxida-

tion of the specimen. The specimens were subjected to a heating rate of  $20 \text{ K min}^{-1}$  from room temperature to 1273 K followed by isothermal holding at 1273 K for 30 min and subsequently cooled down to room temperature at  $20 \text{ K min}^{-1}$  or  $140 \text{ K min}^{-1}$ .

The pushrods applied were made of polycrystalline  $\text{Al}_2\text{O}_3$  or quartz (see Fig. 1). The thermocouple 1 which controls the applied temperature program was fixed at the surface at half height of the cylindrically shaped specimen. The longitudinal temperature gradient was measured by spot welding two further thermocouples, one at each end (L and R) of the specimen. The corresponding temperatures measured by these thermocouples are  $T_{\text{centre}}$  and  $T_{\text{end(L)}}$  and  $T_{\text{end(R)}}$ . Pt–Pt<sub>90</sub>Rh<sub>10</sub> thermocouples were used. It was verified that the same temperature gradient holds for both halves of the specimen (i.e. the temperature profile is symmetric with respect to the central cross-sectional plane of the specimen).  $T_{\text{end(L)}}$  and  $T_{\text{end(R)}}$ , differ only about 0.5 K, whereas the difference of  $T_{\text{centre}}$  and  $T_{\text{end(L)}}$  or  $T_{\text{end(R)}}$  amounts to almost 5 K (see Fig. 2).

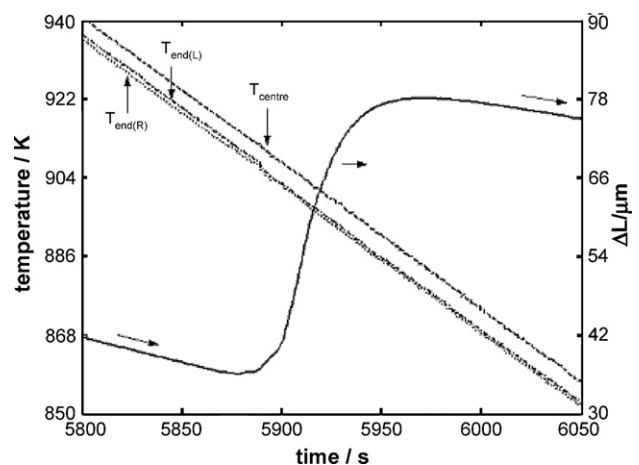


Fig. 2. The measured temperatures,  $T_{\text{centre}}$ ,  $T_{\text{end(L)}}$ ,  $T_{\text{end(R)}}$ , and the length change,  $\Delta L$ , as a function of time during the  $\gamma \rightarrow \alpha$  transformation of Fe–5.91 at.% Ni cooled at  $20 \text{ K min}^{-1}$ .  $T_{\text{end(L)}}$ ,  $T_{\text{end(R)}}$  are the temperatures measured by thermocouples spot welded on the left and right ends of the specimen, respectively.

Table 1  
Chemical composition of the iron and nickel used (mass in ppm)

Element	Fe	Ni
C	11	23
Si	13	0.23
Cu	1	0.18
Ti	0.6	5.3

For reproducible temperature measurements for different specimens, involving different spot weldings, a reproducible spot welding procedure had to be applied. The oxide free surface of the specimens should exhibit the same surface finish (roughness) and the spot welding should provide a mechanically stable connection. The thermocouple wires were spot welded separately parallel to each other. This procedure needed only a small electric current and short time for the spot welding. Thus, the dissolution of elements of the specimen in the thermocouple wire could be minimized. The calibrated temperature exhibits an overall uncertainty of about  $\pm 2$  K (for a single experiment this is a systematic error).

### 3. Experimental results

The obtained length change after calibration as a function of temperature ( $T_{\text{centre}}$ ) for Fe–5.91 at.% Ni during cooling at  $20 \text{ K min}^{-1}$  from 1273 K is shown in Fig. 3. The enlarged portion of the length change during the  $\gamma \rightarrow \alpha$  transformation as a function of time as well as temperature is shown in Fig. 4. The length change as a function of temperature shows considerable fluctuations during the transformation as compared to the length change as a function of time because of small fluctuations in the temperature. The measured values for the length change,  $\Delta L$  and for the temperatures,  $T_{\text{centre}}$  and  $T_{\text{end}}$ , have been plotted as function of cooling time,  $t$ , during the  $\gamma \rightarrow \alpha$  transformation for the two applied cooling rates in Fig. 5(a) and (b). The difference between  $T_{\text{centre}}$  and  $T_{\text{end}}$  reveals that a temperature gradient occurs in the specimen during cooling. The small fluctuations in the  $T_{\text{centre}(t)}$  and  $T_{\text{end}(t)}$  data (measured locally at the surface for both cooling rates), which are in the range of  $\pm 0.5$  K inside the transformation range and  $\pm 0.2$  K outside the transformation range, do not represent the temperature in the specimen: Temperature fluctuations of  $T_{\text{end}}$  in the transformation range are, at least partially, due to a locally changing heat release due to successions of periods of acceleration and retardation in

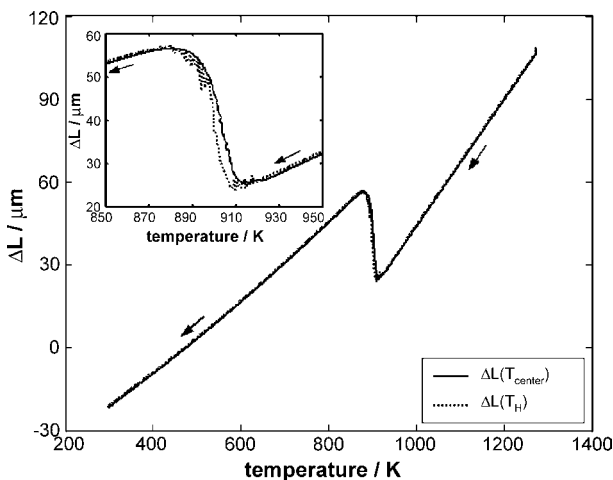


Fig. 3. The length change,  $\Delta L$ , as a function of  $T_{\text{centre}}$  and the homogeneous temperature,  $T_{\text{H}}$  (after incorporating the correction procedure) of Fe–5.91 at.% Ni cooled at  $20 \text{ K min}^{-1}$  from a temperature of 1273 K. The enlarged inset shows the  $\Delta L$  as a function of  $T_{\text{centre}}$  and  $T_{\text{H}}$  during  $\gamma \rightarrow \alpha$  transformation.

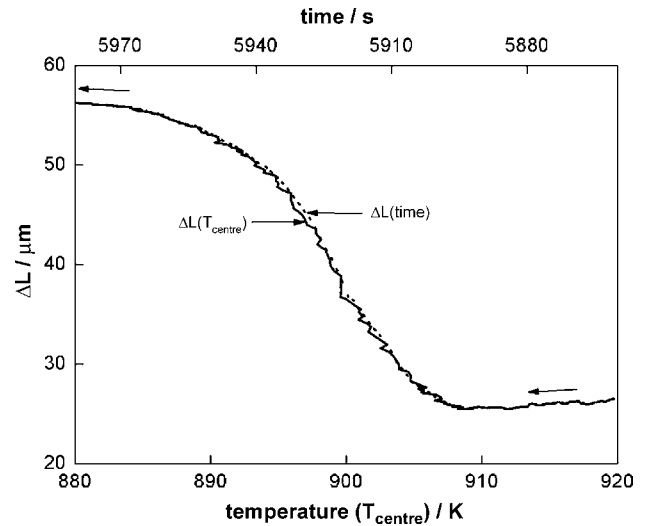


Fig. 4. The length change,  $\Delta L$ , as a function of  $T_{\text{centre}}$  (solid line) and time (dashed line) upon  $\gamma \rightarrow \alpha$  transformation at a cooling rate of  $20 \text{ K min}^{-1}$  of Fe–5.91 at.% Ni. The arrows are the guidelines for the eyes along the direction of cooling.

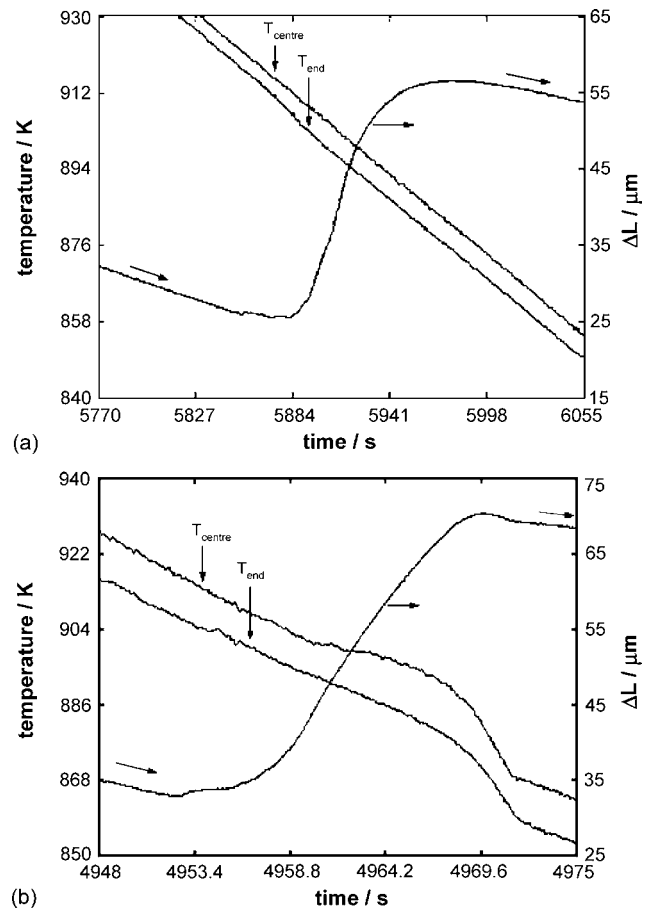


Fig. 5. The length change,  $\Delta L$  and the temperatures,  $T_{\text{centre}}$  and  $T_{\text{end}}$ , as a function of time during the  $\gamma \rightarrow \alpha$  transformation of Fe–5.91 at.% Ni for the two applied cooling rates: (a)  $20 \text{ K min}^{-1}$  and (b)  $140 \text{ K min}^{-1}$ .

the prevailing interface-controlled ( $\gamma \rightarrow \alpha$ ) transformation process, in correspondence with observations by in situ transmission electron microscopy analysis [5] and dilatometry [6]. The fluctuations of  $T_{\text{centre}}$  are smaller because thermocouple1 (Fig. 1; measures the temperature,  $T_{\text{centre}}$ ) controls the applied temperature program; however, the program cannot compensate local temperature fluctuations in the range of  $\pm 0.2$  K. Against this background the temperatures  $T_{\text{centre}}(t)$  and  $T_{\text{end}}(t)$  have been smoothed by applying a moving weighted-average filter [7] (see Fig. 6).

The smoothed  $T_{\text{centre}}$  and  $T_{\text{end}}$  values decrease about linearly with time/temperature. The temperature difference ( $T_{\text{centre}} - T_{\text{end}}$ ) is practically constant until the onset of the  $\gamma \rightarrow \alpha$  transformation, but different for both applied cooling rates ( $T_{\text{centre}} - T_{\text{end}}$ ) is larger for the cooling rate of  $140 \text{ K min}^{-1}$ ; Fig. 6). During the phase transformation  $T_{\text{end}}$ , for the cooling rate of  $20 \text{ K min}^{-1}$ , and both  $T_{\text{centre}}$  and  $T_{\text{end}}$ , for the cooling rate of  $140 \text{ K min}^{-1}$ , decrease nonlinearly with temperature, which is ascribed to heat release during the phase transformation (recalescence). The temperature difference ( $T_{\text{centre}} - T_{\text{end}}$ ) as function of time, at  $0.5 \text{ K}$  intervals, during

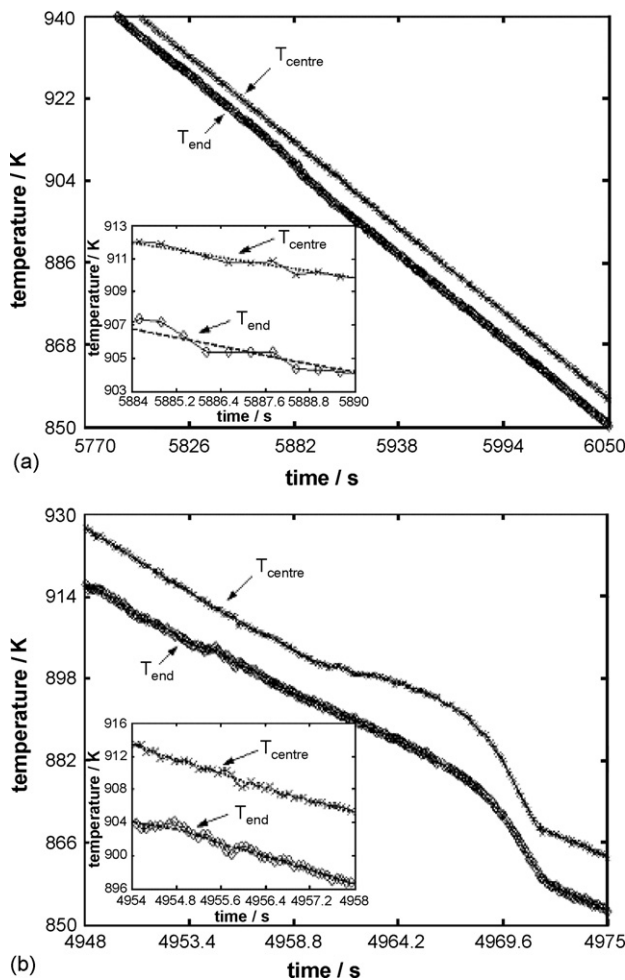


Fig. 6. The calibrated (data points) and smoothed (dashed and dotted lines) temperatures,  $T_{\text{centre}}$  and  $T_{\text{end}}$  as a function of time during the  $\gamma \rightarrow \alpha$  transformation of Fe–5.91 at.% Ni for the two applied cooling rates: (a)  $20 \text{ K min}^{-1}$  and (b)  $140 \text{ K min}^{-1}$ .

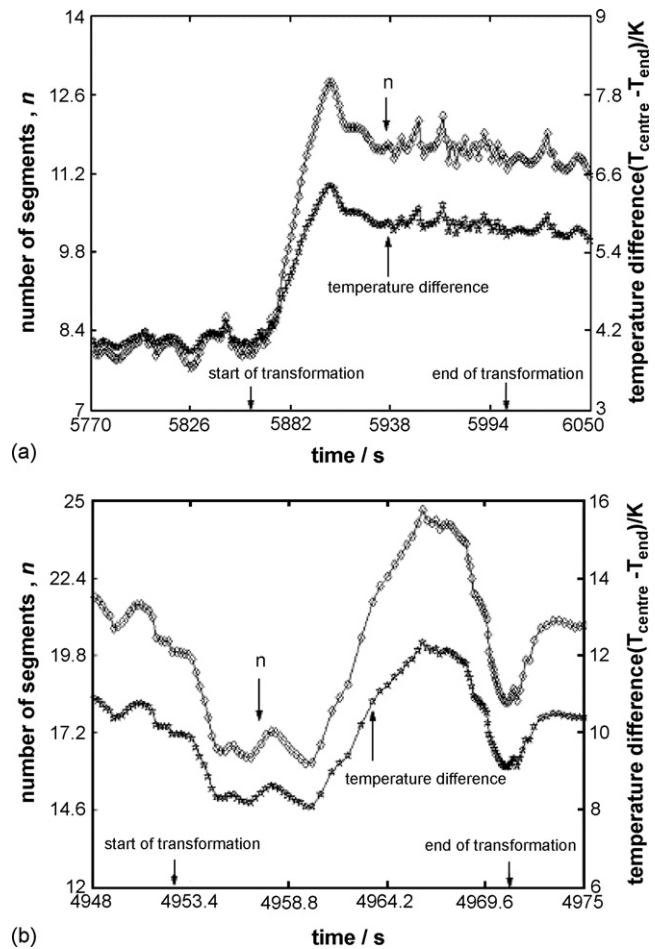


Fig. 7. The temperature difference ( $T_{\text{centre}} - T_{\text{end}}$ ), and the corresponding number of segments,  $n$  (for a temperature difference of two adjacent segments,  $\Delta T$ , equal to  $0.5 \text{ K}$ ; cf. Eq. (1)) as function of time during the  $\gamma \rightarrow \alpha$  transformation of Fe–5.91 at.% Ni for two applied cooling rates: (a)  $20 \text{ K min}^{-1}$  and (b)  $140 \text{ K min}^{-1}$ .

the  $\gamma \rightarrow \alpha$  transformation, is shown in Fig. 7. The temperature difference ( $T_{\text{centre}} - T_{\text{end}}$ ) increases during the first part of the transformation for the cooling rate of  $20 \text{ K min}^{-1}$  and remains nearly constant for the largest part of the transformation, whereas for the cooling rate of  $140 \text{ K min}^{-1}$  the temperature difference oscillates between the temperature difference at the start of about  $10 \text{ K}$  and at the end of transformation of about  $9 \text{ K}$  (cf. Fig. 7(a) and (b)).

#### 4. Correction procedure for temperature inhomogeneity

##### 4.1. Transformation dilation correction

A temperature gradient exists in the longitudinal direction of the dilatometry specimen undergoing induction heating/cooling. In order to calculate the dilation of the specimen as a function of a homogeneous temperature, the entire specimen in the longitudinal direction is hypothetically divided (cut perpendicular to the length axis) into a number of small specimens, which will be called *segments*. Each segment is assigned a homogeneous temperature. If the length change contribution by a segment can

be calculated at each homogeneous temperature then the length change for the entire specimen at the same homogeneous temperature follows from a simple addition of the contributions of all segments.

The temperature profile can be assumed to be linear from the centre (=half height) of the specimen, where the temperature is identical to the measured value of  $T_{\text{centre}}$ , to the end of the specimen, where the temperature is identical to the measured value of  $T_{\text{end}}$  (see Figs. 1 and 6). Further, the temperature profile is taken symmetrical with respect to centre, where  $T_{\text{centre}}$  is measured (this symmetry has been verified; Fig. 2 and see Section 2). The number of segments of one half of the specimen is given by the chosen temperature difference,  $\Delta T$  of two adjacent segments as

$$n = \frac{T_{\text{centre}} - T_{\text{end}}}{\Delta T} \quad (1)$$

where  $n$  is a real number ( $n = j + \eta$ ,  $j = 1, 2, \dots$  (i.e. integer),  $0 \leq \eta < 1$ ) and  $\Delta T \ll T_{\text{centre}} - T_{\text{end}}$ . Thus, there are  $j$  full segments and one fractional segment (fraction is  $\eta$ ) in one half of the specimen. The entire specimen is composed of  $2n$  segments along the longitudinal direction;  $n$  from the centre of the specimen to each of both ends.

The obtained numbers of segments along with the temperature differences ( $T_{\text{centre}} - T_{\text{end}}$ ) are shown for  $\Delta T = 0.5$  K and two different applied cooling rates in Fig. 7. The temperature difference ( $T_{\text{centre}} - T_{\text{end}}$ ) for the applied cooling rate of  $20 \text{ K min}^{-1}$  increases from about 4 K to about 6 K during the first part of the transformation (see Section 3) and hence the obtained number of segments,  $n$ , increases from about 8 to about 12 (see Fig. 7(a)). With the cooling rate of  $140 \text{ K min}^{-1}$  the temperature difference varies from about 9 K to about 10 K during the transformation and thus the obtained number of segments,  $n$  varies from about 18 to about 20 (see Fig. 7(b)).

The length of a full segment,  $l$  in the specimen at a particular time during cooling/heating is

$$l = \frac{L/2}{n} \quad (2)$$

where  $L$  is the length of the specimen at the time considered and  $n = j + \eta$ . There are  $j$  full segments with length  $l$  and one fractional segment with a length  $l_{\eta} = \eta l$ . The above described segmentation is performed during the cooling/heating at different times dictated by  $T_{\text{centre}}$  where the difference between the successive values of  $T_{\text{centre}}$ ,  $\Delta T_{\text{step}}$ , is taken equal to the constant, chosen temperature difference  $\Delta T$  governing the segmentation (cf. Eq. (1)). It is important to note that as the transformation proceeds the number of segments and hence the segment length changes.

From the experimentally determined length changes, due to thermal expansion/shrinkage and transformation, first the length change due to the transformation as function of an operative temperature, either  $T_{\text{centre}}$  or  $T_{\text{end}}$  (cf. Fig. 8(a)), has to be determined. To this end the tangents AB and CD have been drawn in Fig. 8(a) and (b) as a function of time. They represent the (extrapolated) length change of the austenitic specimen upon cooling (AB) and the (extrapolated) length change of the (fully

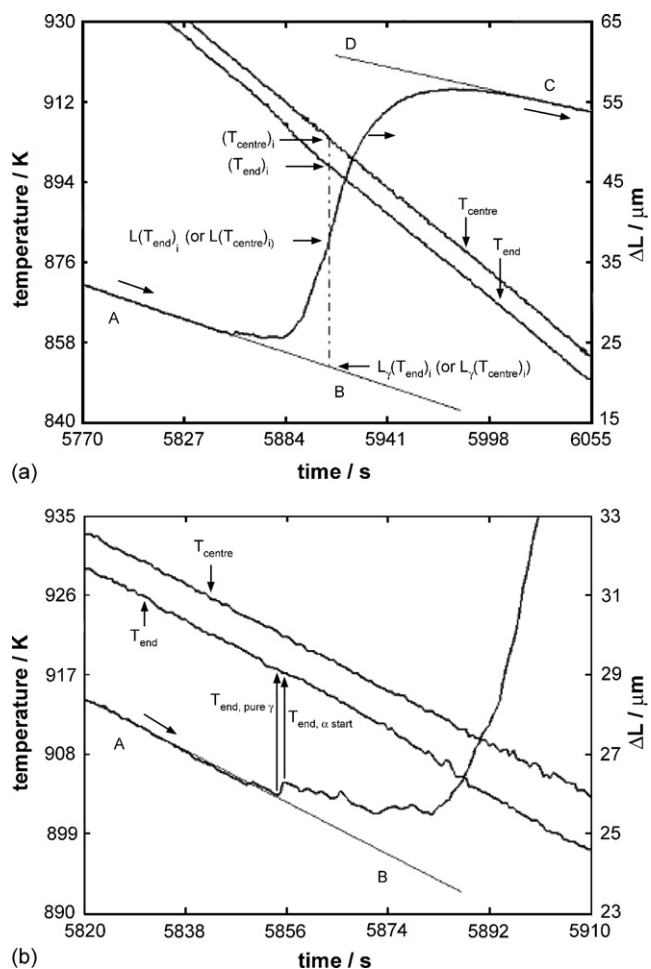


Fig. 8. (a) The length change,  $\Delta L$  as a function of time during the  $\gamma \rightarrow \alpha$  transformation of Fe-5.91 at.% Ni for the cooling rate of  $20 \text{ K min}^{-1}$ . AB and CD represent the (extrapolated) thermal shrinkage of the  $\gamma$  phase and the (extrapolated) thermal expansion of the  $\alpha$  phase and (b) enlarged portion of (a) around the start of the transformation where  $T_{\text{end, pure } \gamma}$  and  $T_{\text{end, } \alpha \text{ start}}$  correspond to the temperatures at the end of the stability of the pure  $\gamma$  phase and at the first observable deviation of length change from the linear shrinkage of the pure  $\gamma$  phase.

transformed) ferritic specimen upon cooling (DC). The first deviations from the tangents AB and CD, for decreasing temperature and increasing temperature, respectively, determine the value of the start and end temperatures of the transformation. The length change  $\Delta L$  due to the transformation is here given as a function of  $T_{\text{end}}$ :

$$\Delta L(T_{\text{end}})_i = L(T_{\text{end}})_i - L_{\gamma}(T_{\text{end}})_i \quad (3)$$

where  $(T_{\text{end}})_i$  denotes the temperature at the end of the specimen (in the longitudinal direction) at (time)/temperature step  $i$  during transformation,  $L(T_{\text{end}})_i$  is the length of the transforming specimen at  $(T_{\text{end}})_i$  and  $L_{\gamma}(T_{\text{end}})_i$  is the (extrapolated) length of the pure austenitic specimen at  $(T_{\text{end}})_i$  as obtained by extrapolation according to AB.

Recognizing that the lowest temperature in the specimen of inhomogeneous temperature is  $T_{\text{end}}$ , the length change for the entire specimen during transformation can be discussed as follows. For the temperature-step  $i = 1$ , i.e. at the onset of the

transformation, the number of *transforming* (fractional) segments,  $n_{\text{start}}$  can be determined from the value measured for  $T_{\text{end}}$  at the end of the stability of pure  $\gamma$  phase (just before the first deviation from linear part AB),  $T_{\text{end,pure } \gamma}$ , and the value measured for  $T_{\text{end}}$  corresponding to the first observable deviation from the linear part AB pertaining to the pure  $\gamma$  phase,  $T_{\text{end},\alpha \text{ start}}$  according to (cf. Eq. (1)):

$$n_{\text{start}} = \frac{T_{\text{end,pure } \gamma} - T_{\text{end},\alpha \text{ start}}}{\Delta T} \quad (4)$$

$T_{\text{end},\alpha \text{ start}}$  is measured for the moment where the first deviation of  $T_{\text{end}}$  (larger than the scatter of the data) from the linear thermal dilation (shrinkage) behaviour of the pure  $\gamma$  phase is recorded as a value of  $T_{\text{end}}$  (see Fig. 8(b)). The (fractional) segment transforming in the first temperature step is shown as a shaded segment in Fig. 9(a) (one at each end of the specimen). The observed length change,  $\Delta L(T_{\text{end}})_{i=1}$  (see Fig. 8(b)) corresponds to transformation in the above two fractional segments. Because the difference in temperature between adjacent segments,  $\Delta T$  is taken equal to  $\Delta T_{\text{step}}$  constant (cf. below Eq. (2)), for the next temperature step ( $i=2$ ) the observed length change,  $\Delta L(T_{\text{end}})_{i=2}$  is due to transformation in the last two segments at both ends of the specimen (shown in shades). Thus, the total length change due to the transformation in temperature steps

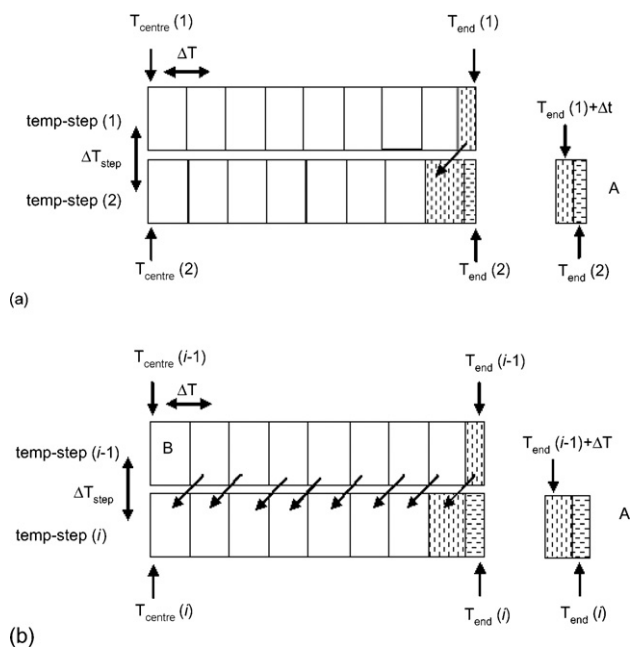


Fig. 9. A hypothetical schematic presentation of segmentation of the specimen experiencing a ( $\gamma \rightarrow \alpha$ ) transformation. In each case (a and b) the segmentation shown in the upper box belongs to temperature step  $i-1$  and the one shown in the bottom box belongs to temperature step  $i$ . The arrows connect pairs of segments with the same temperature in two successive temperature steps. (a) Initial two temperature steps ( $i=1$  and 2) where only the shaded segments transform. (b) Two consecutive, intermediate temperature steps where all segments transform.  $T_{\text{centre}} - T_{\text{end}} = \text{constant}$  or the difference is small and hence the numbers of full segments in temperature steps  $i-1$  and  $i$  are the same. Part A shown at the right in (a) and (b) shows the so-called excess segments (see text); which, together with segment B, are responsible for the change of specimen length occurring upon proceeding from temperature step  $i-1$  to temperature step  $i$ :  $\Delta L(T_{\text{end}}) - \Delta L(T_{\text{end}})_{i-1}$  (see text and Eq. (5)).

1 and 2 is given by the sum of (a) the length change contribution by the shaded (fractional) segment in temperature step  $i=1$ ,  $\Delta L(T_{\text{end}})_{i=1}$  (segments in Fig. 9 connected by the arrow have the same temperature) and (b) the *excess* segments contributing to the length changes  $\Delta L(T_{\text{end}})_{i=2}$  in temperature step  $i=2$ : the (fractional) end segment plus part of the segment adjacent to it (if in temperature step  $i=1$  the end segment was fractional). The *excess* segments, shown in part A in Fig. 9(a), are responsible for the increase in length ( $\Delta L(T_{\text{end}})_{i=2} - \Delta L(T_{\text{end}})_{i=1}$ ), in temperature step  $i=2$  as compared to temperature step  $i=1$ . As the transformation proceeds, further segments contribute to the length change due to transformation.

Consider two successive intermediate temperature steps  $i-1$  and  $i$ . At both temperature steps all the segments are transforming. The top box in Fig. 9(b) represents the segments in the specimen at temperature step  $i-1$  and the box underneath represents the segments in the specimen at temperature step  $i$  of the transformation. For a small or no change in temperature gradient, the temperatures of those segment pairs of temperature steps,  $i-1$  and  $i$  connected by arrows in Fig. 9(b) are equal, and hence their contributions to specimen length change are equal. Then upon increasing  $i$  ( $i=1, 2, 3, \dots$ ), the length change contribution of the end segments at each step  $i$  follows from the measured values of  $\Delta L(T_{\text{end}})_i$  by subtracting the length change contributions of the other segments, which can be derived from the determined values for the length change contributions of  $T_{\text{end}}(i)$  values occurring at the previous temperature steps.

The temperatures and the length change contributions from pairs of segments connected by the arrows are identical (Fig. 9(b)). So, the resulting increase in length in temperature step  $i$  as compared to temperature step  $i-1$  originates from (a) the excess segments in temperature step  $i$  (as defined above), indicated by part A in Fig. 9(b) and (b) segment B in temperature step  $i-1$  adjacent to the centre of the specimen. (Note: for the case shown in Fig. 9(a) segment B has not been considered because at the onset of transformation it does not contribute to any length change.) Now, in both the cases (Fig. 9(a) and (b)) the two fractional (excess) end segments, contained in part A, can be treated as one segment with the same length but of homogeneous temperature  $T_{\text{end}}(i)$  provided  $\Delta T$  is sufficiently small. The contribution of the uncanceled (see the arrows in Fig. 9(b) and text above) segment B in Fig. 9(b) in temperature step  $i-1$  is known because a segment of temperature B has already occurred as an end segment (called here B') at some temperature step before. Although the temperature of both these segments (B in temperature step  $i-1$  and B' in some previous temperature step) are the same, the corresponding segment lengths need not be necessarily equal: the segment length  $l$ , depends on  $L$  and  $n$  (see Eq. (2)) which both change during the transformation (cf. above discussion and Fig. 7). Hence, a multiplication factor,  $\mu$  is introduced, which accounts for the fractional increase or decrease in length of a full segment B in temperature step  $i-1$  as compared to the length of the corresponding segment B' of the same temperature in a temperature step  $i-k$ .

On the basis of entire discussion above the length change of the entire specimen in temperature step  $i$ , as compared to temperature step  $i-1$ , is then directly related to the difference in

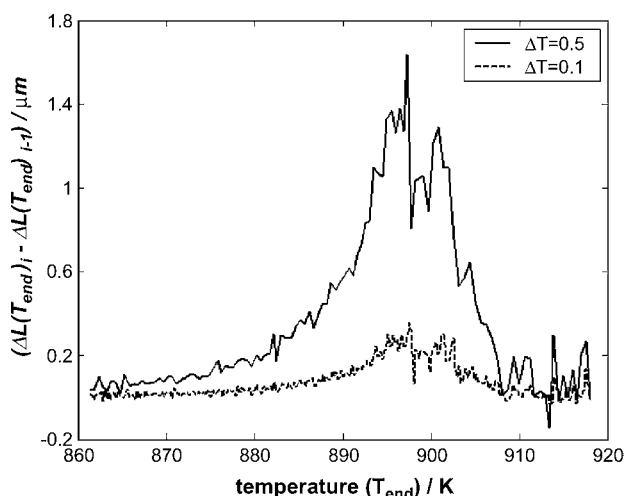


Fig. 10. Calculated length change,  $\Delta L(T_{\text{end}}) - \Delta L(T_{\text{end}})_{i-1}$ , as function of temperature  $T_{\text{end}}$  during the  $\gamma \rightarrow \alpha$  transformation for the indicated cooling rate.

length change contribution from part (“segment”) A and segment B. Recognizing that segment B’ occurred as end “segment” A (=part A) in temperature step  $i - k$  one can now write

$$\Delta L(T_{\text{end}})_i - \Delta L(T_{\text{end}})_{i-1} = 2[\Delta L_{A,i}(T_{\text{end}})_i - \mu \Delta L_{A,i-k}(T_{\text{end}})_{i-k}] \quad (5)$$

where  $\Delta L_{A,i}(T_{\text{end}})_i$  and  $\Delta L_{A,i-k}(T_{\text{end}})_{i-k}$  are the length change contributions from the end “segment” A (part A) in temperature step  $i$  and in temperature step  $i - k$ , respectively. The factor 2 recognises that the segments part A and B occur twice at temperature steps  $i$  and  $i - 1$ , respectively.

The difference (length change),  $\Delta L(T_{\text{end}})_i - \Delta L(T_{\text{end}})_{i-1}$  is shown in Fig. 10 for the cooling rate of  $20 \text{ K min}^{-1}$  for two different  $\Delta T$  ( $\Delta T = 0.1 \text{ K}$  and  $0.5 \text{ K}$ ). The difference upon cooling initially increases reaches a maximum and subsequently decreases. Because for a  $\Delta T = 0.1 \text{ K}$  five times more segments

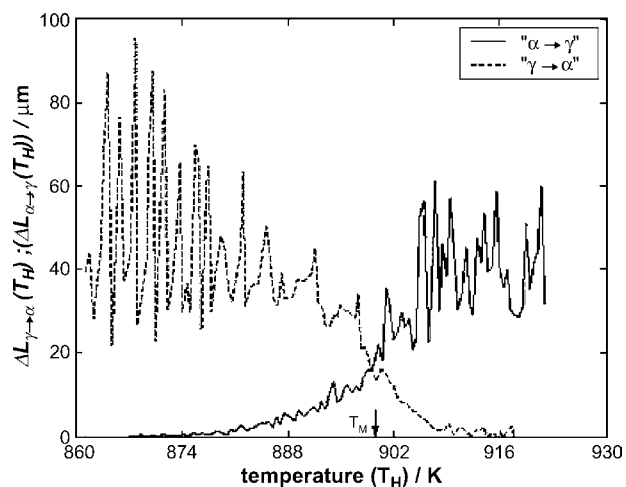


Fig. 11. The length change,  $\Delta L_{\gamma \rightarrow \alpha}(T_H)$  and  $\Delta L_{\alpha \rightarrow \gamma}(T_H)$  as function of homogeneous temperature,  $T_H$ , during the  $\gamma \rightarrow \alpha$  transformation starting from untransformed prior  $\gamma$  phase to end with fully transformed  $\alpha$  phase and the (hypothetical)  $\alpha \rightarrow \gamma$  transformation using the dilatational data measured upon cooling ( $\gamma \rightarrow \alpha$ ) starting the fully transformed  $\alpha$  phase to end with untransformed prior  $\gamma$  phase. Cooling (hypothetical heating) rate:  $20 \text{ K min}^{-1}$ .

occur than for a  $\Delta T = 0.5 \text{ K}$ , the scatter for both cases should be compared after multiplication of the length changes for a  $\Delta T = 0.1 \text{ K}$  with this factor of 5. Then it follows that the scatter in the data is larger for  $\Delta T = 0.1 \text{ K}$  than for  $\Delta T = 0.5 \text{ K}$ . From these data, and because  $\mu$  and  $\Delta L_{A,i-k}(T_{\text{end}})_{i-k}$  are known, the unknown value of  $\Delta L_{A,i}(T_{\text{end}})_i$  can be determined for all  $i$  in a recursive way (Eq. (5)).

Hence, the above treatment leads to the determination of the change of length of the end “segment” (part A) for all values of  $(T_{\text{end}})_i$ . Because the absolute length of the end “segment” A at all temperature steps  $i$  is also known, the relative change of length of the “segment” A at the temperature  $(T_{\text{end}})_i$  is known and thus the absolute length change of a hypothetical full segment at  $(T_{\text{end}})_i$  is known as well. On this basis the length change of the entire specimen at a homogeneous temperature  $(T_{\text{end}})_i$  as compared to the specimen length at  $(T_{\text{end}})_{i-1}$  is given by

$$\Delta L_{\gamma \rightarrow \alpha}(T_H) = \frac{L}{l} \Delta L_{\text{end}}(T_H) \quad (6)$$

where  $\Delta L_{\text{end}}$  denotes the length change at  $T_H (= (T_{\text{end}})_i)$  for a (hypothetical) full end segment,  $L$  the length of the specimen at the time considered, and  $l$  is the length of a full segment.

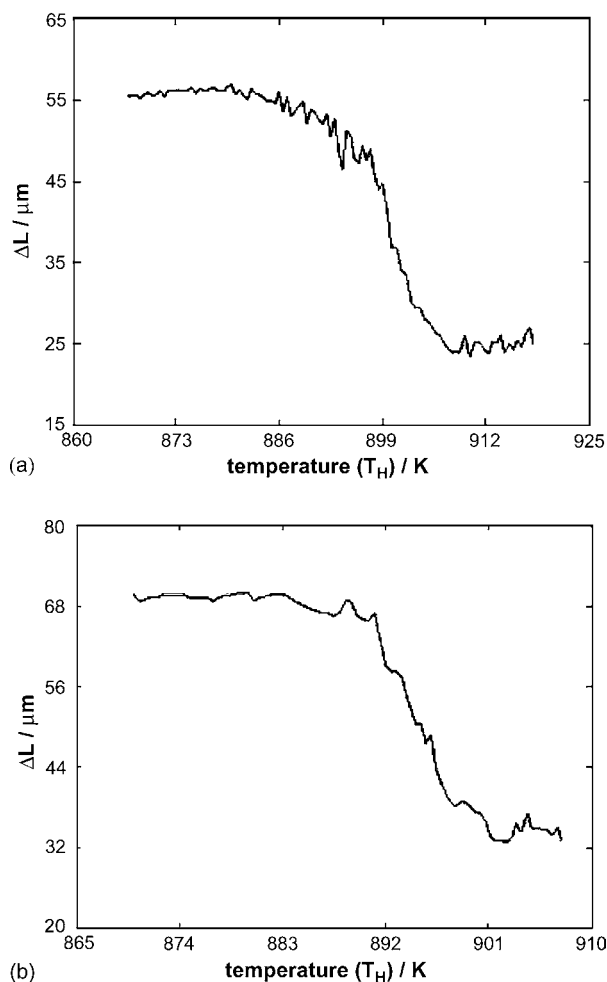


Fig. 12. The calculated (corrected for temperature inhomogeneity) length change,  $\Delta L$ , as function of homogeneous temperature,  $T_H$ , pertaining to the  $\gamma \rightarrow \alpha$  transformation for two applied cooling rates: (a)  $20 \text{ K min}^{-1}$  and (b)  $140 \text{ K min}^{-1}$ .

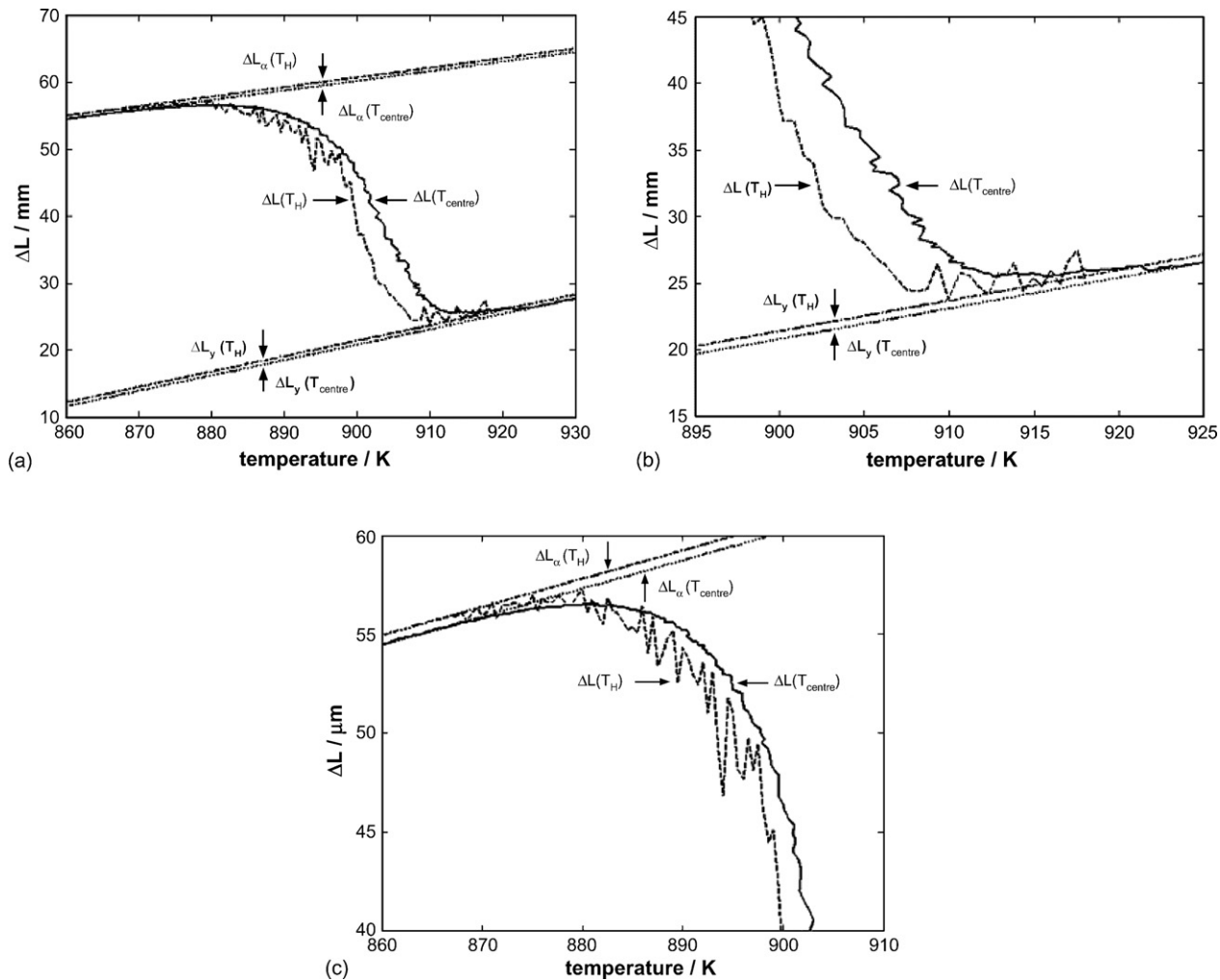


Fig. 13. (a) The calibrated,  $\Delta L(T_{\text{centre}})$ , and corrected,  $\Delta L(T_H)$ , dilations of the transforming specimen ( $\gamma \rightarrow \alpha$  transformation of Fe–5.91 at.% Ni for the applied cooling rate of  $20 \text{ K min}^{-1}$ ) along with the measured and corrected dilations of the pure austenite phase and the pure ferrite phase,  $\Delta L_\gamma$  and  $\Delta L_\alpha$ , as a function of temperature. (b) and (c) Enlargements of parts of (a) (see text for discussion).

The thus obtained length change  $\Delta L_{\gamma \rightarrow \alpha}(T_H)$  from Eq. (6), for the cooling rate of  $20 \text{ K min}^{-1}$  is shown in Fig. 11. A considerable fluctuation is imposed on the resulting curve. Obviously, the difference term,  $\Delta L(T_{\text{end}})_i - \Delta L(T_{\text{end}})_{i-1}$  is very sensitive to the experimental fluctuation in  $\Delta L(T_{\text{end}})$  in particular near the end of transformation where the change of length  $\Delta L(T_{\text{end}})$  eventually becomes of the order of the experimental fluctuation.

The dilation correction procedure was executed also using the same dilatational data but starting at the fully transformed  $\alpha$  phase to end with untransformed prior  $\gamma$  phase.<sup>1</sup> The resulting hypothetical  $\Delta L_{\alpha \rightarrow \gamma}(T_H)$  in Fig. 11 shows a similar behaviour as  $\Delta L_{\gamma \rightarrow \alpha}(T_H)$  with fluctuations of increasing amplitude towards the end of the (hypothetical) transformation. To remedy the occurrence of these fluctuations the following procedure was adopted. First a suitable intermediate temperature,  $T_M$ , in the temperature range of the transformation was chosen

beyond which the fluctuations become large (see the temperature  $T_M$  indicated with the arrow in Fig. 11). Secondly, the resulting length change,  $\Delta L_{\gamma \rightarrow \alpha}(T_H)$  (“ $\gamma \rightarrow \alpha$ ”; Fig. 11) until  $T_M$  was added to the extrapolated linear shrinkage of the parent  $\gamma$  phase (after the correction as described in Section 4.2) and the resulting length change  $\Delta L_{\alpha \rightarrow \gamma}(T_H)$  (“ $\alpha \rightarrow \gamma$ ”; Fig. 11) until  $T_M$  was subtracted from the extrapolated linear expansion of the product  $\alpha$  phase (after the correction as described in Section 4.2). The thus obtained final results for two applied cooling rates are shown in Fig. 12. Indeed, by this procedure the scatter on the dilatation curve has been decreased substantially.

#### 4.2. Thermal dilation correction for a not transforming phase

The measured (extrapolated) lengths for both the parent and the product phases,  $L_\gamma(T_{\text{centre}})$  and  $L_\alpha(T_{\text{centre}})$  for pure  $\gamma$  and  $\alpha$ , have to be corrected as well for the nonhomogeneous temperature of the specimen; i.e. the lengths of the pure parent specimen and the pure product specimen have to be calculated for a hypothetical specimen of homogeneous temperature,  $T_H$ .

<sup>1</sup> The highest temperature of the specimen is  $T_{\text{centre}}$  at any temperature step. Then, upon virtual heating the first segment to transform is given by temperature,  $T_{\text{centre}}$ . So, the homogeneous temperature in this case is given by  $T_H = T_{\text{centre}}$  (cf. discussion of the procedure given for cooling).



Taking  $T_H$  as  $T_{\text{centre}}$ , the length of all the segments with temperature different from  $T_{\text{centre}}$  (i.e. lower than  $T_{\text{centre}}$ ) have to be extended with contributions proportional to  $l_s \alpha (T_{\text{centre}} - T_s)$  where  $l_s$  denotes the length of the segment (i.e. for a full segment  $l_s = l$  and for a fractional segment  $l_s = l_\eta$  (see discussion of Eq. (2))),  $T_s$  is the segment temperature and  $\alpha$  is the thermal expansion coefficient. Hence, the length of the specimen with homogeneous temperature  $T_H (=T_{\text{centre}})$  is given by for pure austenite:

$$L_\gamma(T_H) = L_\gamma(T_{\text{centre}}) + \frac{L/2}{n} \left[ 2 \left( \alpha_\gamma \left( \sum_{i=1}^j i \Delta T \right) + \alpha_\gamma (\eta \Delta T) \right) \right] \quad (7)$$

for pure ferrite:

$$L_\alpha(T_H) = L_\alpha(T_{\text{centre}}) + \frac{L/2}{n} \left[ 2 \left( \alpha_\alpha \left( \sum_{i=1}^j i \Delta T \right) + \alpha_\alpha (\eta \Delta T) \right) \right] \quad (8)$$

where  $\alpha_\gamma$  and  $\alpha_\alpha$  are the linear thermal expansion coefficients of austenite and ferrite, respectively. Note that, in contrast with the treatment in Section 4.1, for cooling/heating of a pure phase which is not undergoing a phase transformation,  $T_{\text{centre}} - T_{\text{end}}$  and thus  $n$  is practically constant. The segment length,  $l_s$  (for full and fractional segments) can then also be considered to be practically constant.

On the basis of Eqs. (7) and (8) and using the appropriate values of  $L$ ,  $n$ ,  $\alpha_\gamma$  and  $\alpha_\alpha$  (in preparation) and  $\Delta T$ , the dilation corrections for pure  $\gamma$  and  $\alpha$  phases,  $L_\gamma(T_H) - L_\gamma(T_{\text{centre}})$  and  $L_\alpha(T_H) - L_\alpha(T_{\text{centre}})$ , can be determined straightforwardly. Results are shown in Table 2. The thus determined dilation corrections for the two phases have to be applied to the measured linear dilations of the respective phases (AB and CD in Fig. 8(a)) as a function of the temperature  $T_{\text{centre}}$ .

#### 4.3. Full dilatation correction

Finally, the length of the specimen as a function of homogeneous temperature  $T_H$  in the transformation range of the  $\gamma \rightarrow \alpha$  transformation is obtained by adding the corrected dilation,  $\Delta L_{\gamma \rightarrow \alpha}(T_H)$ , pertaining to the  $\gamma \rightarrow \alpha$  transformation (see Sec-

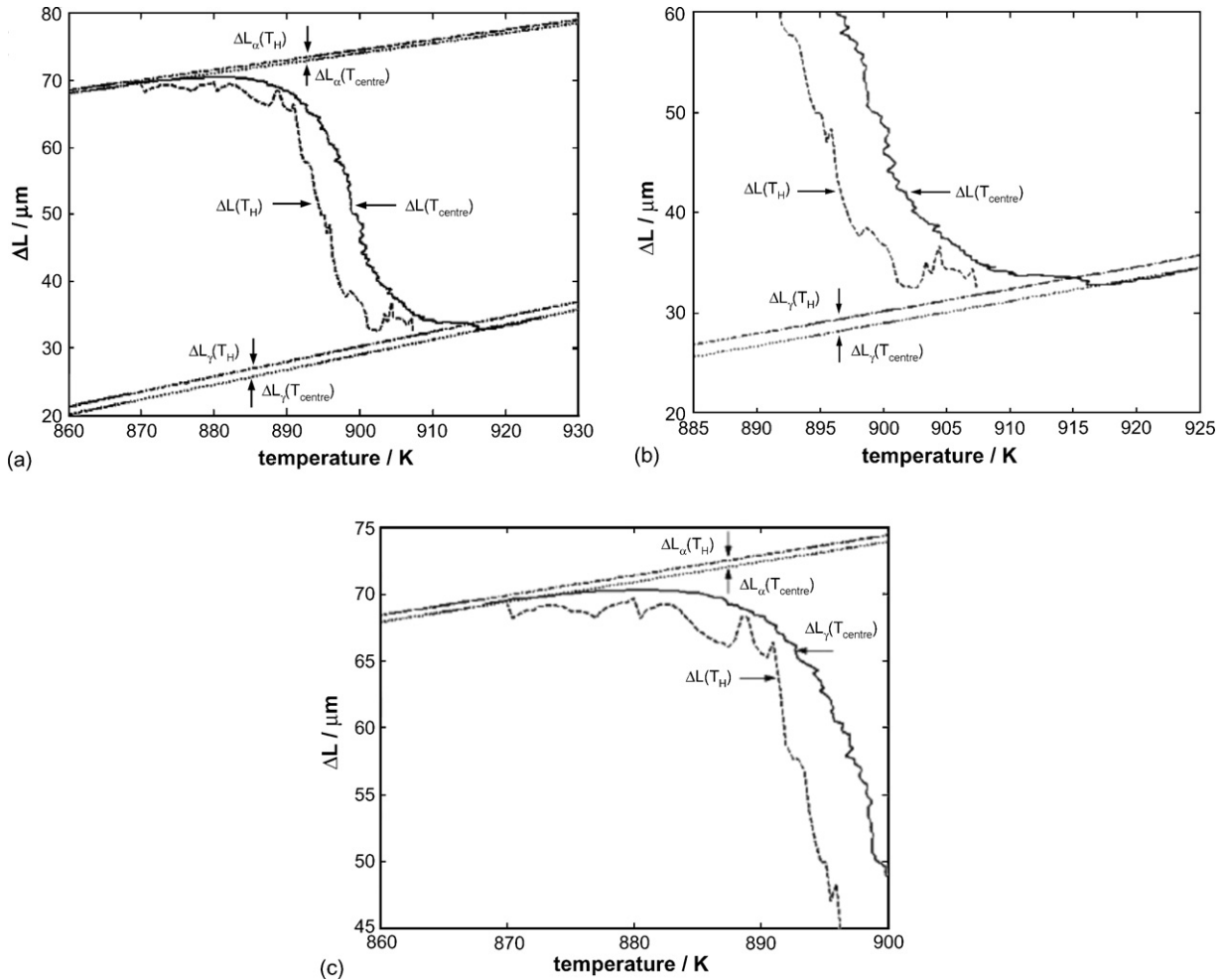


Fig. 14. (a) The calibrated,  $\Delta L(T_{\text{centre}})$ , and corrected,  $\Delta L(T_H)$ , dilation of the transforming specimen ( $\gamma \rightarrow \alpha$  transformation of Fe-5.91 at.% Ni for the applied cooling rate of  $140 \text{ K min}^{-1}$ ) along with the measured and corrected dilations of the pure austenite phase and the pure ferrite phase,  $\Delta L_\gamma$  and  $\Delta L_\alpha$ , as a function of temperature. (b) and (c) Enlargements of parts of (a) (see text for discussion).

Table 2

The applied cooling rates, number of segments at the onset of the  $\gamma \rightarrow \alpha$  transformation,  $n_\gamma$ , the number of segments at the end of the  $\gamma \rightarrow \alpha$  transformation,  $n_\alpha$  and the length change correction for the pure  $\gamma$  phase,  $L_\gamma(T_H) - L_\gamma(T_{\text{centre}})$  and pure  $\alpha$  phase,  $L_\alpha(T_H) - L_\alpha(T_{\text{centre}})$

Cooling rate (K min <sup>-1</sup> )	$n_\gamma$	$n_\alpha$	$L_\gamma(T_H) - L_\gamma(T_{\text{centre}})$ ( $\mu\text{M}$ )	$L_\alpha(T_H) - L_\alpha(T_{\text{centre}})$ ( $\mu\text{M}$ )
20	4.2	5.6	0.6	0.6
140	10.0	8.5	1.3	0.8

tion 4.1) to the corrected thermal dilation of the pure  $\gamma$  phase,  $L_\gamma(T_H)$  (see Section 4.2), as a function of homogeneous temperature,  $T_H$ . The resulting length change as function of  $T_H$  is shown in Fig. 12 for both applied cooling rates of 20 K min<sup>-1</sup> and 140 K min<sup>-1</sup> for the Fe–5.91 at.% Ni specimens. The as measured dilations as function of  $T_{\text{centre}}$  and the corrected dilations as a function of  $T_H$  can be compared for applied cooling rates of 20 K min<sup>-1</sup> and 140 K min<sup>-1</sup> in Figs. 13 and 14. Evidently, the correction is larger for 140 K min<sup>-1</sup> than for 20 K min<sup>-1</sup>, because the specimen subjected to higher cooling rate experiences a larger temperature gradient. The dilation corrections for the pure phases ( $\gamma$  and  $\alpha$ ) and for the specimen that transforms are independent of each other. Then it is satisfying to observe (see the enlargement of parts of Figs. 13(a) and 14(a) in Figs. 13(b) and (c) and 14(b) and (c), respectively, for the applied cooling rates of 20 K min<sup>-1</sup> and 140 K min<sup>-1</sup>) that the corrected length change due to the  $\gamma \rightarrow \alpha$  transformation matches well with both the thermal shrinkage of the pure  $\gamma$  phase at the start of the transformation and with the thermal shrinkage of the pure  $\alpha$  phase at the end of the transformation. So the correction procedures proposed in Sections 4.1 and 4.2 are compatible with each other.

## 5. Conclusions

1. A distinct temperature gradient occurs along the longitudinal direction of an inductively heated dilatometer specimen dur-

ing heating and/or cooling, which increases with increasing heating/cooling rate. Thus, the measured dilation cannot be interpreted directly in terms of transformation kinetics.

2. For the first time a temperature correction procedure was developed to obtain the dilation as a function of a homogeneous temperature. The method is based on hypothetical segmentation of the specimen into a number of small segments in the longitudinal direction so that each segment can be supposed to have a homogeneous temperature and such that the temperature difference between adjacent segments in a particular temperature step and the temperature difference of successive temperature steps are identical. The dilation contribution from one segment, during transformation, is calculated from the difference in dilation for the whole specimen between two successive temperature steps. This leads to a recursive procedure to calculate the relative change of length during the transformation. The obtained dilation for one segment can then be used to calculate the dilation for the full specimen.
3. Combining the *independent* dilation corrections for (i) temperature inhomogeneity in the not transforming parent and product phases and for (ii) the temperature inhomogeneity during the transformation leads to length changes for the specimen which are compatible.

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