

Calibration of the differential dilatometric measurement signal upon heating and cooling; thermal expansion of pure iron

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Received 5 September 2003; received in revised form 7 October 2003; accepted 11 October 2003

Abstract

Differential dilatometry is a technique for precise measurements of the thermal dilatation of materials. A procedure has been presented for calibration of the differential dilatometric measurement signal (length change as a function of temperature), both upon heating and cooling. Measurements of the thermal expansion of pure iron up to 1223 K have been performed by a high-resolution differential dilatometer (resolution in length measurement of about 10 nm). The magnetic contribution to the length change has been introduced for the first time in an analytic expression for the linear thermal expansion coefficient of ferritic iron. The dilatation signal has been calibrated on heating and cooling using a sapphire specimen for which recommended dilatation data are available. The ferro- to paramagnetic transition (characterized by the Curie point temperature) has been adopted successfully to calibrate the temperature in dilatometric measurements upon heating and cooling. © 2003 Elsevier B.V. All rights reserved.

Keywords: Differential dilatometer; Calibration; Heating; Cooling; Linear thermal expansion; Curie temperature

1. Introduction

Many solid materials exhibit structural changes, e.g. phase transformations, upon changing the temperature. These phase transformations are usually accompanied by a significant change in specific volume. The change in volume of a solid material is usually measured by the corresponding change in length of a specimen of this material. Thus, measurements of the change in length of solid materials are often applied for the determination of the kinetics of phase transformation of metals and alloys (e.g. [1–6]). If, upon increasing or decreasing the temperature, a phase transformation does not occur, the length of the specimen changes by thermal dilatation. Thereby, the linear thermal expansion coefficient, i.e. the relative length change divided by the corresponding temperature interval, is an important thermophysical property.

Many different methods for measuring the dilatation of solids (due to temperature change) have been developed. The

methods for measuring the dilatation of a specimen may be divided into two classes [7,8]: (1) *relative* methods in which the length change of a specimen of the material concerned is measured relative to the length change of a specimen of another, reference material, using, e.g. a push-rod dilatometer; and (2) *absolute* methods, in which the expansion of specimen of the material concerned is directly measured, using, e.g. a laser interferometric dilatometer. The relative dilatometers possess a length change resolution in the range 10–2000 nm which, for the best ones, is still somewhat lower than that of absolute dilatometers. (For the best laser interferometric dilatometers, the length change resolution may be as small as about 5 nm [9].) The complicated operation of the absolute methods limits their use [10]. Here, the focus is on the use of a relative dilatometer, which is widely used because of its relatively simple construction, the ease of handling and the large adaptability to shapes of the samples to be investigated. Relative dilatometry, in particular because of its nowadays possible, high length change resolution of about 10 nm, is an extremely powerful tool to measure phase transformation kinetics.

Differential dilatometry involves the recording of the thermal dilatation of a sample relative to that of a reference material [10]. The two factors that limit the accuracy of the determination of the length change by differential dilatom-

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etry are: (i) the thermal dilatation behavior of the sample holder and the push-rods, which is normally accounted for by the baseline of the dilatometer and (ii) the accuracy of the temperature of the specimen, because the corresponding thermocouple is usually located between the sample investigated and the reference specimen, and thus is not in contact with the specimen to be investigated.

Little attention has been paid on the calibration of relative dilatometers in the past years. To the authors' knowledge, only two references [11,12] deal with the calibration of the dilatation signal on heating and cooling and of the temperature signal on heating of relative dilatometers. Here, a new and much more accurate calibration method of a differential dilatometric measurement signal upon heating and cooling has been proposed. The dilatation of pure iron has been used as an example for providing absolute values of length change upon temperature change. For the first time, the magnetic contribution to the (linear) thermal expansion coefficient of pure iron has been introduced in the quantitative analytic expression for thermal dilatation of ferritic iron. The Curie temperature of pure iron and nickel has been taken as a standard for the absolute temperature calibration during heating and cooling experiments.

2. Experimental

The differential dilatometer DIL 802 (Bähr-Thermoanalyse GmbH) was used to measure the thermal dilatation behavior of iron. With this instrument the difference in length change of the sample to be investigated and an inert reference sample is measured. The measuring head is thermally stabilized and insensitive to mechanical vibration. The measurements were performed under flowing high purity argon (7.01h^{-1}) to avoid oxidation of the specimen. A schematic diagram of the differential push-rod dilatometer is given in Fig. 1. The furnace consists of a SiC heater and a water-cooled jacket. The two push rods are single-crystal aluminum-oxide (sapphire), which are positioned inside the inner aluminum-oxide tube. The inner aluminum-oxide tube is placed in a vacuum tight outer aluminum-oxide tube, which is connected to the measuring head (see Fig. 1). The achievable measurement temperature range is from room

Table 1
Chemical composition of the iron used

Element	Content
C	14
Si	13
Cu	1
Ti	0.6
Fe	Balance

Unit: ppm in mass, as provided by Aldrich Chemical Company.

temperature to 1823 K. The temperature of the sample and of the reference material was measured with a Pt₉₀Rh₁₀–Pt thermocouple placed in between the sample and the reference specimen. The displacement measurement device to detect changes in length between the sample and reference specimen is a linear variable differential transformer (LVDT) positioned in the measuring head, which is maintained at 298.15 ± 0.1 K with cooling water using a thermostat.

The employed iron, supplied by Aldrich Company, is a rod with a diameter of 6.3 mm. The purity of the iron has been indicated in Table 1. The as-received pure Fe rods were hammered down to a rod with a diameter of about 5.5 mm. In order to achieve a homogeneous microstructure, the rods were sealed in a quartz container filled with argon gas at 2×10^4 Pa, and annealed at 1473 K for 100 h. Thereafter, the rods were machined into dilatometric specimens with a diameter of 5 mm and a length of 10 mm.

3. Calibration

In differential dilatometric measurements, the temperature and displacement signals are measured as a function of time. These values are determined by a thermocouple near the sample investigated and the LVDT in the measurement head, respectively.

The displacement measured with the LVDT consists of the relative change in length of the sample against that of the reference specimen and the net effect of additional changes in length of parts of the dilatometer subjected as well to temperature changes by the furnace, such as the push rods and the inner aluminum-oxide tube. The effect of the additional changes in length can be determined by performing a

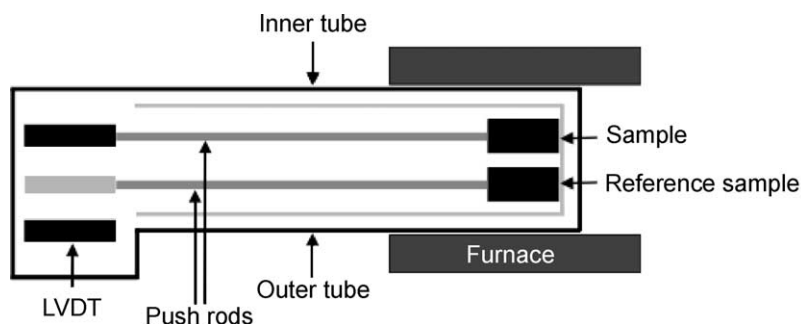


Fig. 1. Schematic diagram of the differential dilatometer DIL 802 from Bähr Company.

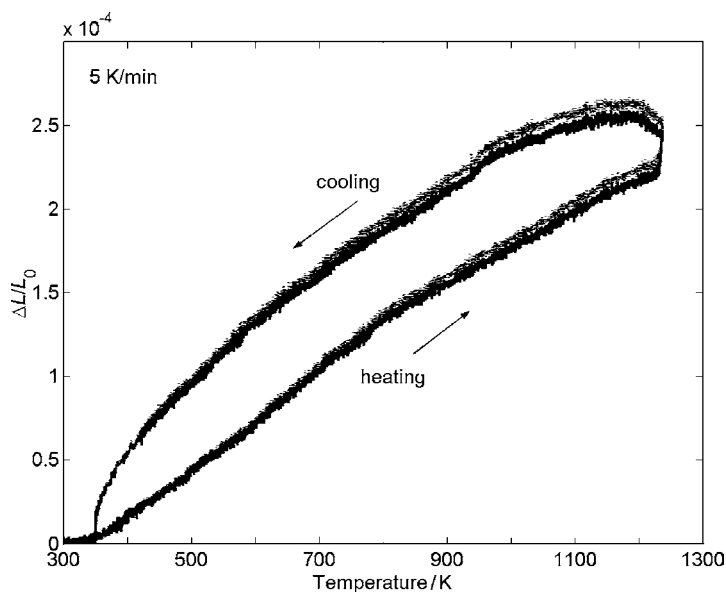


Fig. 2. Recorded relative length changes of polycrystalline Al_2O_3 (as sample) against polycrystalline Al_2O_3 (reference) during continuous heating (5 K min^{-1}) from room temperature to 1223 K and subsequent continuous cooling (5 K min^{-1}) interrupted by an isothermal annealing at 1223 K for 30 min for three successive heat treatment cycles.

measurement with two samples of the same dimensions and made from the same material. The recorded relative length change ($\Delta L/L_0$, L_0 is the sample length at 293.15 K and ΔL is the temperature dependent length change (difference) recorded by LVDT) of a polycrystalline aluminium oxide (Al_2O_3) sample against a polycrystalline Al_2O_3 'reference' specimen, during continuous heating (5 K min^{-1}) from room temperature to 1223 K and subsequent continuous cooling (5 K min^{-1}) interrupted by an isothermal annealing at 1223 K for 30 min, is shown in Fig. 2. The recorded small change in length as function of temperature reflects the difference in length change of both push rods and the effect of any inhomogeneity in the change in length of the inner aluminum-oxide tube (e.g. due to anisotropy of the microstructure of the inner tube). The results shown in Fig. 2 thereby represent the baseline of the differential dilatometer for the chosen heat treatment procedure. It also has to be emphasized that different holding time at high temperature would largely change the corresponding baseline.

The reproducibility of the baseline was verified by repeating this measurement. The recorded relative length changes of polycrystalline Al_2O_3 against polycrystalline Al_2O_3 for two additional successive heat treatment cycles are also shown in Fig. 2. The variation in $\Delta L/L_0$ of the instrumental baseline is less than at most about 5×10^{-6} , implying a very good reproducibility. This value could be compared with the relative length change due to, for example, the transformation of austenite to ferrite in pure iron which is associated with a value of $\Delta L/L_0$ of about 4×10^{-3} (cf. Fig. 6a). It has to be remarked that a proper sample installation and the surface quality of the specimen and of the push rods especially at the contacting area (front side of the specimen and

the push rods) play a crucial role in achieving this reproducibility.

Calibration of the displacement (length) signal is performed by measuring the length change of a sapphire (sample) against the length change of polycrystalline Al_2O_3 (reference). The measured $\Delta L/L_0$ signal expresses (i) the baseline of the instrument (see above) and (ii) the difference in length change between the sapphire and the polycrystalline Al_2O_3 specimen.² The difference between these measured $\Delta L/L_0$ data and the recommended (reference) values for the dilatation of the used sapphire specimen serves as a calibration (correction) of the displacements ($\Delta L/L_0$ values) recorded in measurement runs performed with the same heat treatment procedure.

The temperature measured with a thermocouple is generally different from that of the specimen to be investigated, because the thermocouple is not in direct contact with the sample. The temperature calibration depends not only on temperature but also on the heating or cooling rate. The most widely used temperature calibration method is based on the melting of pure substances: by comparison of the measured and the known, literature values of the melting temperatures, a temperature correction is determined. The superheating of a solid upon heating, before melting occurs, can be ignored, but undercooling of the liquid upon cooling, before solidification takes place, is unavoidable. Therefore,

² Note that this length change is due to the anisotropy of the thermal expansion of single-crystal Al_2O_3 (=sapphire); the polycrystalline Al_2O_3 (=reference) will show a more or less macroscopically isotropic thermal expansion. Thus, the observed length change depends on the crystal orientation of the sapphire crystal used, with respect to the dilatometer axis.

the solidification of pure molten substances is not suitable for the temperature calibration during cooling. To each heating rate to be used, a separate temperature calibration has to be performed or inter- or extrapolation of calibration parameters with respect to heating rate has to be performed [13].

It was recently proposed to adopt the Curie temperature corresponding to the ferro- to paramagnetic phase transformation for temperature calibration (also) upon cooling. This idea was successfully applied to the calibration of differential thermal analysis (DTA) for determining the heat capacity in heating and cooling experiments [14]. This ferro- to paramagnetic phase transition shows no temperature hysteresis, in contrast to structural transformations, because it involves a transformation during which no material transport takes place [15,16]. The process that governs the transformation, i.e. the change in the relative orientation of electron spins, is, at a temperature far above absolute zero, extremely fast, and thus the Curie temperature is not heating- or cooling-rate dependent. Experimental work on the determination of the Curie temperature of Gd with different heating and cooling rates (spanning a range of five decades in heating and cooling rate) indeed did not reveal any heating- and cooling-rate dependence of the Curie temperature [17]. The Curie temperature of iron and nickel will be used here for the temperature calibration in dilatometry in heating and cooling experiments.

3.1. Calibration procedure

3.1.1. Relative length change

In an ideal differential dilatometric system the measured dilatation is simply the difference in dilatation between that of the specimen to be investigated and the reference speci-

men. However, due to differences in expansion between the two push rods (of sapphire) and the inhomogeneous expansion of the inner polycrystalline Al_2O_3 tube, there is a contribution to the measured expansion, which is called here the baseline of the instrument (see above discussion of Fig. 2). In this work, the known $\Delta L/L_0$ values of a single-crystal Al_2O_3 (sapphire) with orientation of sample axis at $59 \pm 1^\circ$ with respect to the c -axis (supplied by State Center for Measuring Instrument and Certification, Russia, code number 112, certified between 90 and 1800 K) has been used for calibration of the displacement signal as a function of temperature.

Polycrystalline aluminium oxide (Al_2O_3) has always been taken as the reference specimen. The equation expressing the calibration or correction is given by

$$\left(\frac{\Delta L}{L_0}\right)_{\text{cal}} = \left(\frac{\Delta L}{L_0}\right)_{\text{ref}} - \left(\frac{\Delta L}{L_0}\right)_{\text{meas}} \quad (1)$$

with cal denoting the calibration (correction), meas denoting the length change values measured for the single-crystal Al_2O_3 (sapphire) (against polycrystalline Al_2O_3) and ref denoting the known length change data for single-crystal Al_2O_3 specimen. Hence, to obtain the true length change data for an arbitrary specimen, $(\Delta L/L_0)_{\text{cal}}$, determined as described immediately above, has to be added to the measured length change data for an arbitrary specimen (measured against polycrystalline Al_2O_3). Note that $(\Delta L/L_0)_{\text{cal}}$ depends on temperature, the heating/cooling rate, and the holding time at high temperature.

The measured length change of sapphire against polycrystalline Al_2O_3 during the “normal” heat treatment is shown in Fig. 3. The “normal” isochronal treatment is as follows: the specimen was heated from room temperature up to 1223 K (at 5 K min^{-1}) and kept at this temperature for

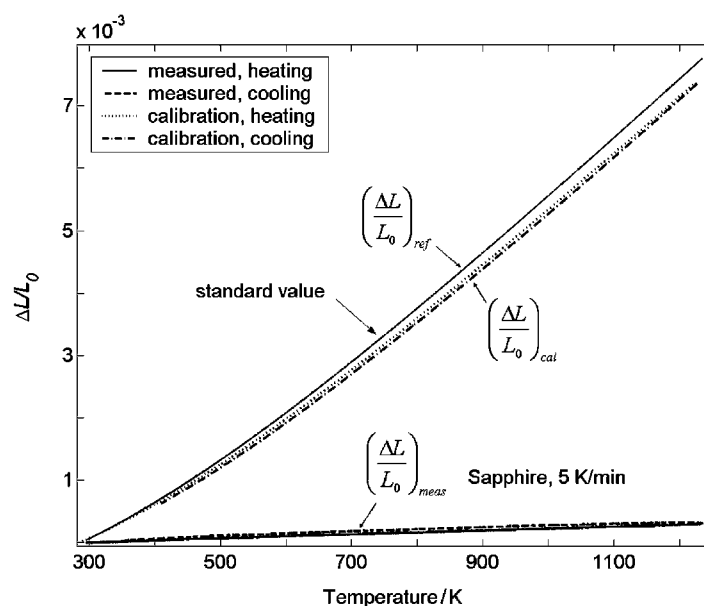


Fig. 3. Comparison of the standard relative length changes, the original measurement results and the calibrations of a sapphire during continuous heating (5 K min^{-1}) from room temperature to 1223 K and subsequent continuous cooling (5 K min^{-1}) interrupted by an isothermal annealing at 1223 K for 30 min.

30 min. Then the specimen was cooled down continuously to 373 K (at 5 K min⁻¹). Comparing Figs. 2 and 3, it follows that the recorded (change of the) length difference between the sapphire specimen and the polycrystalline Al₂O₃ reference specimen is governed by the baseline of the instrument, and apparently not by the difference in length change exhibited by the sapphire (single-crystal Al₂O₃) specimen and the polycrystalline Al₂O₃ specimen. Hence, the difference between $(\Delta L/L_0)_{\text{cal}}$ and $(\Delta L/L_0)_{\text{ref}}$ (see Fig. 3) is predominantly due to instrumental effects and is largely insensitive to the genuine difference in length change of the single-crystal Al₂O₃ specimen and the polycrystalline Al₂O₃ reference specimen.

The difference between the data obtained for heating and cooling (see Figs. 2 and 3) makes clear that different calibrations are required for heating and cooling. By subtracting the measured data from the known $(\Delta L/L_0)_{\text{ref}}$ values of the sapphire, the corresponding calibration correction data $(\Delta L/L_0)_{\text{cal}}$ are obtained for the heating and cooling segments (cf. Eq. (1) and see Fig. 3).

Values for the linear thermal expansion coefficient, $\alpha(T)$, can be calculated from calibrated length change data according to

$$\alpha(T) = \frac{d(\Delta L/L_0)_{\text{cal}}}{dT} \quad (2)$$

To illustrate the sensitivity of values of α for minor scatter in $(\Delta L/L_0)_{\text{cal}}$ values the thermal linear expansion coefficient of the sapphire, directly calculated from the unsmoothed, calibrated heating and cooling data, $(\Delta L/L_0)_{\text{cal}}$, in Fig. 3, is shown in a small temperature range around 775 K in Fig. 4a. The $\alpha(T)$ exhibits a large scatter of $\pm 30\%$ due to small scatter of the $(\Delta L/L_0)_{\text{cal}}$ data (invisible in Fig. 3). This noise originating from the measured $\Delta L/L_0$ data is an unavoidable instrumental effect of relative dilatometry. The scatter in the calculated $\alpha(T)$ can be reduced by using a moving weighted-average filter [18]. The thus resulting $\alpha(T)$ values of the sapphire on heating and cooling are shown in Fig. 4b, which exhibit a scatter of $\pm 8\%$ around 775 K.

3.1.2. Temperature

Several measurements (heat treatment cycles) of $\Delta L/L_0$ applying heating and cooling rates of 5, 10, 15 and 20 K min⁻¹ were performed with pure iron as the sample. The occurrence of the ferromagnetic transition was exhibited as a hump on the length change curve for all applied heating and cooling rates (cf. Fig. 6b) [20]. The linear thermal expansion coefficient of ferritic Fe as determined according to Eq. (2) from the dilatometric measurements at various heating and cooling rates is shown in Fig. 5. The arrows indicate the corresponding apparent temperatures for the Curie temperature for different heating and cooling rates. These apparent Curie point temperatures ($T_{\text{c,a}}$) and the corresponding temperature shifts (ΔT_{c}) with respect to the true Curie temperature of pure iron (1043.0 K) [21] have been listed in Table 2. Clearly, the absolute value of

Table 2

Observed temperature, $T_{\text{c,a}}$, of the ferromagnetic transition temperature as a function of heating and cooling rates and the shift, ΔT_{c} , obtained by comparison of the measured, apparent value and the reference value of the Curie temperature of pure iron (1043.0 K)

Rates (K min ⁻¹)	Heating		Cooling	
	$T_{\text{c,a}}$ (K)	ΔT_{c} (K)	$T_{\text{c,a}}$ (K)	ΔT_{c} (K)
5	1051.3	-8.3	1052.9	-9.9
10	1055.1	-12.1	1049.0	-6.0
15	1058.8	-15.8	1047.4	-4.4
20	1059.7	-16.7	1046.8	-3.8

the temperature shift ΔT_{c} becomes larger upon increasing heating rates, and becomes smaller upon increasing cooling rates. Values of $\Delta T_{\text{c}} \neq 0$ reflect to what extent the sample temperature cannot follow, during heating or cooling, the temperature inside the furnace as measured by the thermocouple. The temperature calibration correction involves adding ΔT_{c} to the temperature values recorded by the thermocouple. To achieve temperature calibration in a temperature range relatively distant from T_{c} of pure iron, other materials with different T_{c} can be applied, as pure nickel with a T_{c} of 631 K [22] (see Section 4.1 and Fig. 7).

4. Thermal expansion of pure iron

4.1. Relative length changes

The relative length changes of pure iron measured during the “normal” heat treatment procedure (cf. Section 3.1.1) are shown in Fig. 6, before and after performance of the calibration correction (i.e. adding $(\Delta L/L_0)_{\text{cal}}$ (cf. Section 3.1.1)). The segment AB of the calibrated curve corresponds to the normal thermal expansion of the specimen during continuous heating in the absence of a phase transformation, part BC represents the $\alpha \rightarrow \gamma$ transformation, during which a length contraction occurs due to the formation of austenite. CD and DE stand for the expansion and contraction of austenite, respectively, upon heating and subsequent cooling (see discussion in Section 4.2.2). Part EF corresponds to the $\gamma \rightarrow \alpha$ reaction, associated with length increase. After completion of the $\gamma \rightarrow \alpha$ transformation the length of the sample decreases continuously down to room temperature due to normal thermal shrinkage (indicated by FG).

In the low temperature part (<573 K), the slope of the recorded relative length changes relatively strongly. This suggests that in this temperature range the specimen to be investigated and the reference specimen cannot follow the programmed temperature at a cooling rate of 5 K min⁻¹. Therefore, a rather low heating and cooling rate (0.08 K min⁻¹) was adopted to measure the thermal expansion of pure iron from ambient temperature to 573 K. The relative length changes of pure iron in the low temperature

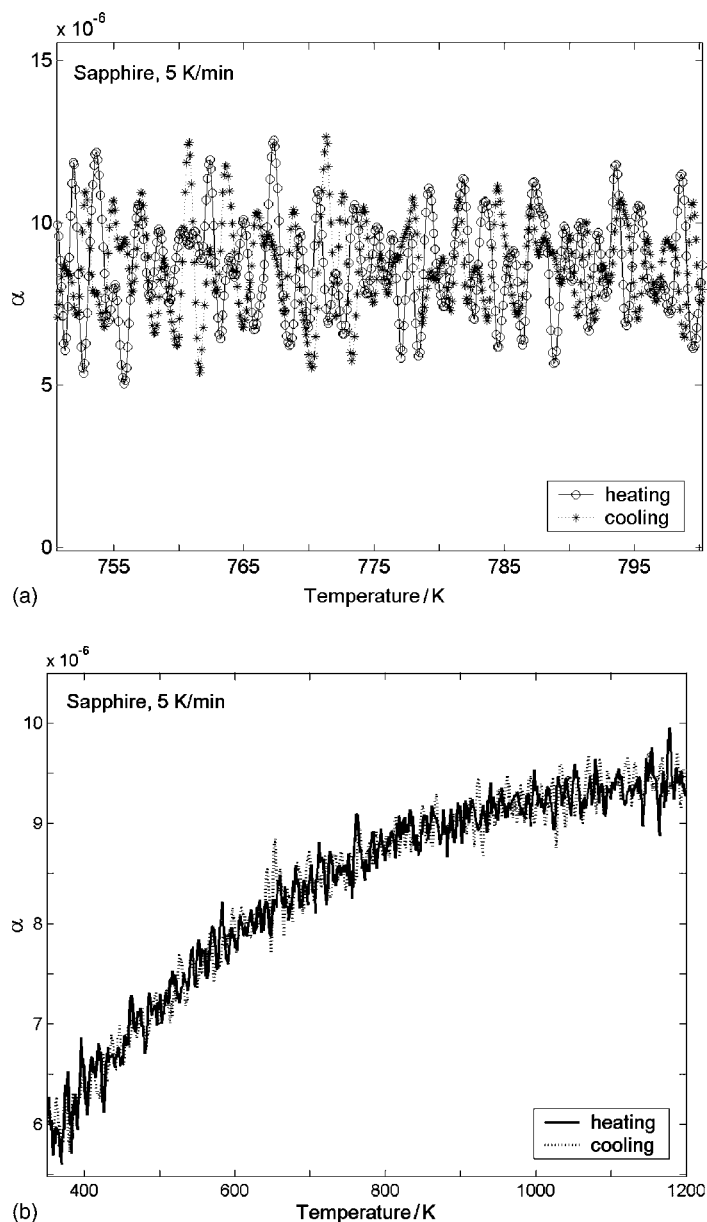


Fig. 4. Thermal linear expansion coefficient, α , of the sapphire during continuous heating (5 K min^{-1}) from room temperature to 1223 K and subsequent continuous cooling (5 K min^{-1}) interrupted by an isothermal annealing at 1223 K for 30 min: (a) using all original data points and (b) filtered results.

range during continuous heating (0.08 K min^{-1}) from room temperature to 573 K and subsequent continuous cooling (0.08 K min^{-1}) interrupted by an isothermal annealing at 573 K for 30 min are shown in Fig. 7 before and after performance of the calibration correction (i.e. adding $(\Delta L/L_0)_{\text{cal}}$ (cf. Section 3.1.1)). The calibration corrected relative length change curves as measured during heating and cooling coincide very well (Fig. 7b), which demonstrates the validity of the *different* calibrations applied to the heating and cooling curves. It is important to remark that the hysteresis-free ferromagnetic transition of pure nickel with a T_c of 631 K [22] was adopted for the temperature calibration in this measurement (cf. Sections 3.1.2 and 4.2.3).

4.2. Linear thermal expansion

4.2.1. Analytical description

An analytical description for the temperature dependence of the relative length change ($\Delta L/L_0$) of pure iron due to thermal expansion can be given by the following equations:

$$\frac{\Delta L}{L_0} = a + bT + cT^2 + dT^3, \quad 300 \text{ K} < T < 1185 \text{ K} \quad (3.1)$$

$$\frac{\Delta L}{L_0} = e + fT + gT^2 + hT^3, \quad 1185 \text{ K} < T < 1225 \text{ K} \quad (3.2)$$

Values of the parameters a to h for pure iron are given in Table 3 [19]. According to [19], these values are considered

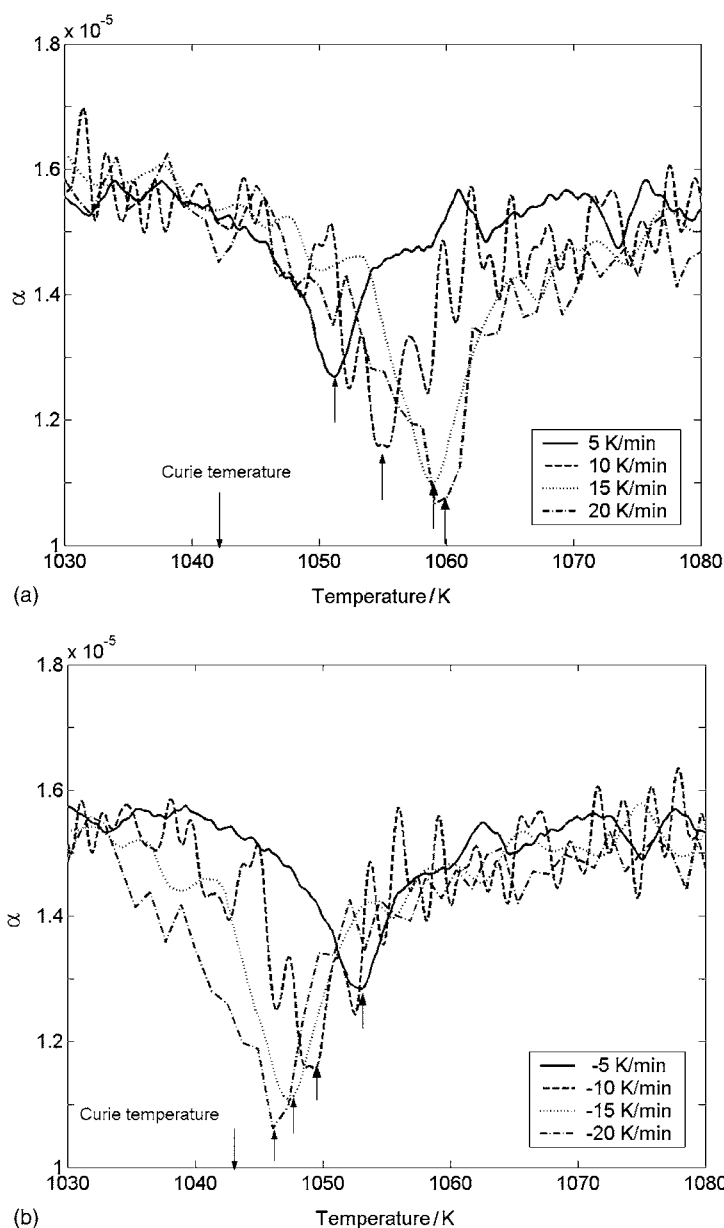


Fig. 5. Linear thermal expansion coefficients (α_v) of ferritic pure iron as determined from the dilatometric measurements at various (a) heating and (b) cooling rates. The Curie temperature and the apparent Curie temperatures are indicated with arrows.

Table 3

Values for parameters in the analytical description (cf. Eqs. (3.1) and (3.2)) of the temperature dependence of the relative length changes of pure iron according to [19]

a	$-2.89\text{E}-3$
b (K^{-1})	$7.35\text{E}-6$
c (K^{-2})	$9.33\text{E}-9$
d (K^{-3})	$-3.14\text{E}-12$
e (K)	$-1.810\text{E}-2$
f (K^{-1})	$2.435\text{E}-5$
g (K^{-2})	$-8.1\text{E}-10$
h (K^{-3})	$2.057\text{E}-13$

to be accurate within $\pm 3\%$ at temperatures below 900 K, within $\pm 5\%$ below 1185 K, and within $\pm 20\%$ above 1185 K.

4.2.2. Linear thermal expansion coefficient of austenite

Consider the high temperature part of the relative length changes in Fig. 6. The $\alpha \rightarrow \gamma$ transformation upon heating is associated with the (inhomogeneous) build up of misfit deformation energy. After completion of the phase change, this misfit strain energy is relaxed which corresponds with a length change. Therefore, the recorded length change curve of austenite during heating is not linear. During the holding at 1223 K for 30 min, the misfit strain energy is fully relaxed (length decrease). Thus, only normal contraction of austenite occurs upon subsequent cooling and the recorded

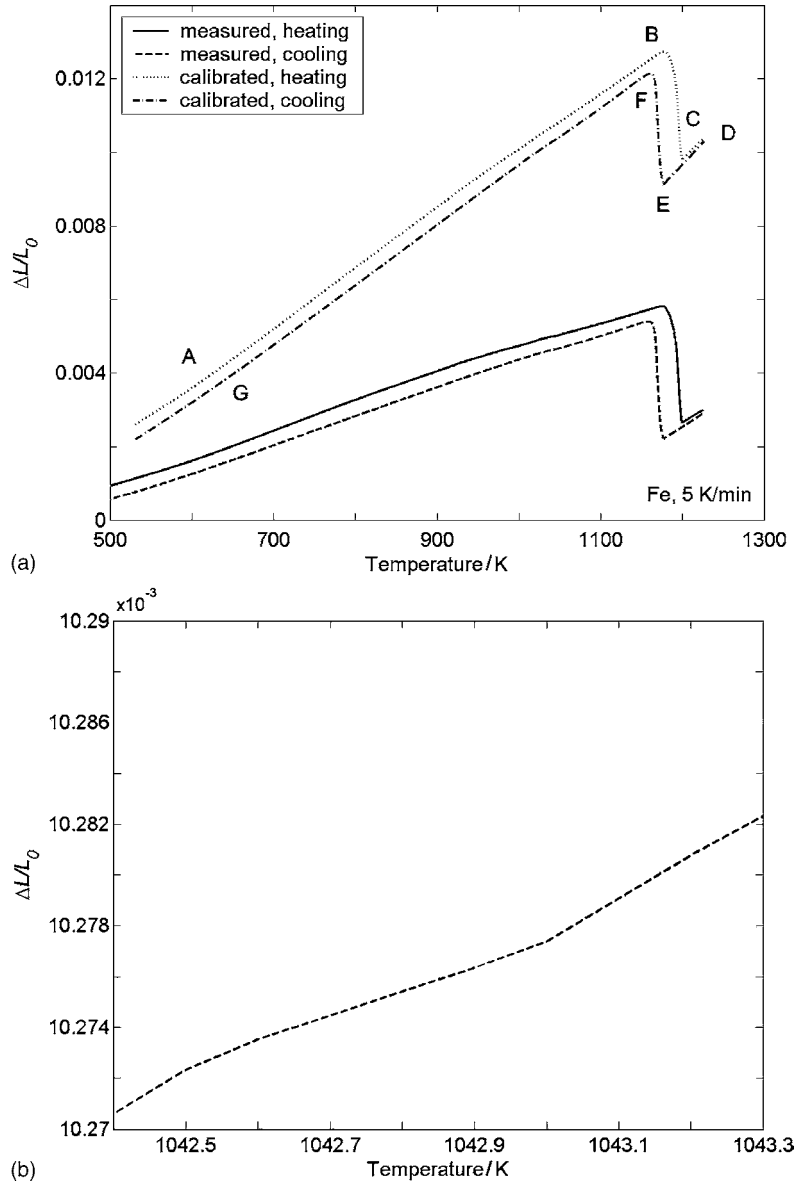


Fig. 6. (a) Comparison of the measured and corrected relative length changes of pure iron during continuous heating (5 K min^{-1}) from room temperature to 1223 K and subsequent continuous cooling (5 K min^{-1}) interrupted by an isothermal annealing at 1223 K for 30 min. (b) Enlargement of the relative length change curve upon heating indicating the magnetic transition.

dilatation data during cooling do reflect the true thermal linear dilatation of the high temperature austenite.

By fitting the determined data of segment DE in Fig. 6 to Eq. (3.2) and using Eq. (2), the linear thermal expansion coefficient of austenitic Fe has been obtained as:

$$\alpha_\gamma = -1.133 \times 10^{-5} + 2.9124 \times 10^{-8}T, \quad (4)$$

1185 K < T < 1225 K

The first constant in Eq. (4) corresponds to the parameter f and the second one to $2g$. A comparison of the thus determined thermal expansion coefficient of austenitic Fe [19] as determined in this work and the one recommended by [19] is presented in Fig. 8.

4.2.3. Linear thermal expansion coefficient of ferrite and temperature calibration

Similarly, as for the heat capacity, the temperature dependence of the thermal linear expansion coefficient of ferrite, α_α , can be described by nonmagnetic and magnet contributions as follows [14]:

$$\alpha_\alpha = b + cT + dT^2 + f_i \exp(e_i T^*) (T^*)^{g_i}, \quad (5)$$

300 K < T < 1185 K

where $T^* = |(T - T_c)/T_c|$, T_c denotes the Curie temperature, b to d are parameters representing the nonmagnetic contribution to α_α (see also Eq. (3.1)), and e_i to g_i are parameters that represent the magnetic contribution to α_α . The label i equals 1 if $T < T_c$, and equals 2 if $T > T_c$.

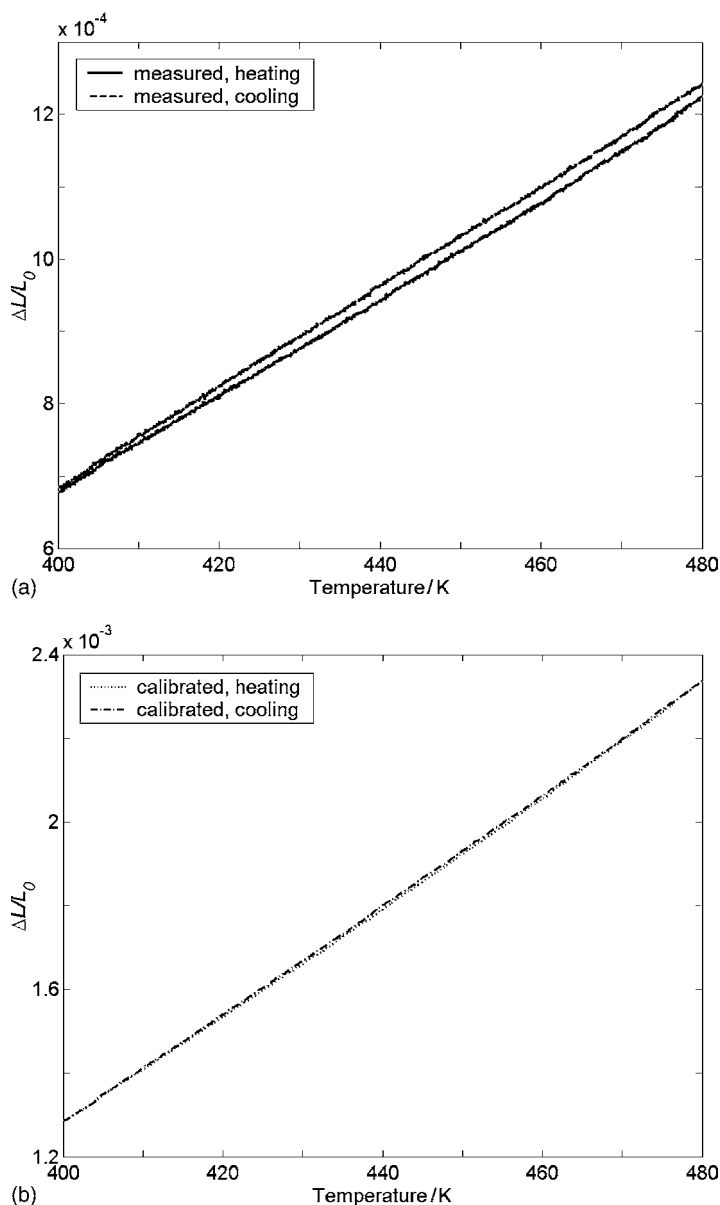


Fig. 7. Comparison of the (a) measured and (b) calibrated relative length changes of pure iron during continuous heating (0.08 K min^{-1}) from room temperature to 573 K and subsequent continuous cooling (0.08 K min^{-1}) interrupted by an isothermal annealing at 573 K for 30 min.

By fitting the linear thermal expansion data of ferritic Fe obtained both upon heating and cooling (the data as shown in Figs. 6 and 7), values for the parameters b to g in Eq. (5) were obtained. The results have been collected in Table 4.

The quality of the fit is very good, as can be seen in Fig. 9 which allows a comparison of the measured (with a heating rate of 0.08 K min^{-1} in the range $350 \text{ K} < T < 550 \text{ K}$ and 5 K min^{-1} in the range $550 \text{ K} < T < 1150 \text{ K}$) and the calculated (Eq. (5)) thermal linear expansion coefficients of ferritic Fe. The measured linear thermal expansion coefficient outside the temperature range of the magnetic transition fits well with the results given in [19] (see Fig. 9). It is clear that the data in [19] cannot at all describe the length change behavior in the temperature range close to and including T_c .

Table 4

Values of the fit parameters for the chosen equation (cf. Eq. (5)) of thermal linear expansion coefficient, α_a , in pure iron

b (K^{-1})	$-4.125\text{E}-9$
c (K^{-2})	$2.100\text{E}-8$
d (K^{-3})	$-1.587\text{E}-11$
e_1	-1.131
e_2	1.920
f_1 (K^{-1})	$1.399\text{E}-5$
f_2 (K^{-1})	$1.210\text{E}-5$
g_1	0.066
g_2	0.033

The parameters e_1 , f_1 and g_1 hold for $T < T_c$, and e_2 , f_2 and g_2 hold for $T > T_c$.

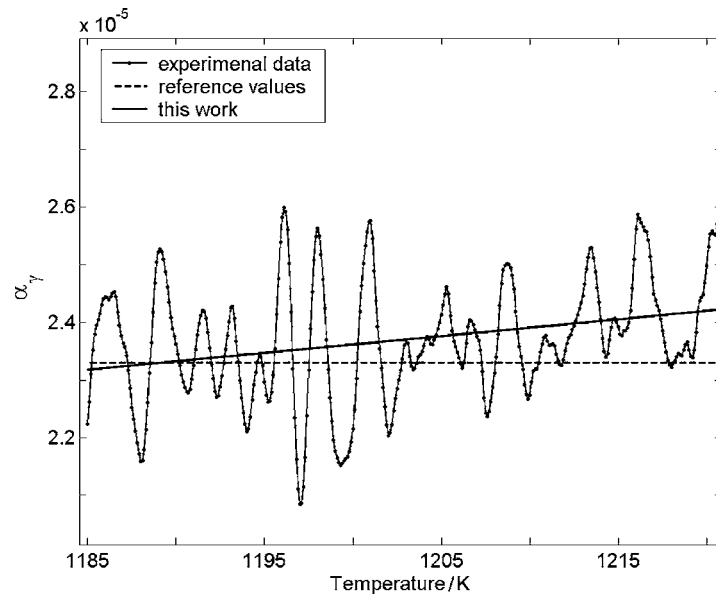


Fig. 8. Comparison of the as-determined, optimized and recommended (by [19]) linear thermal expansion coefficient of austenitic Fe.

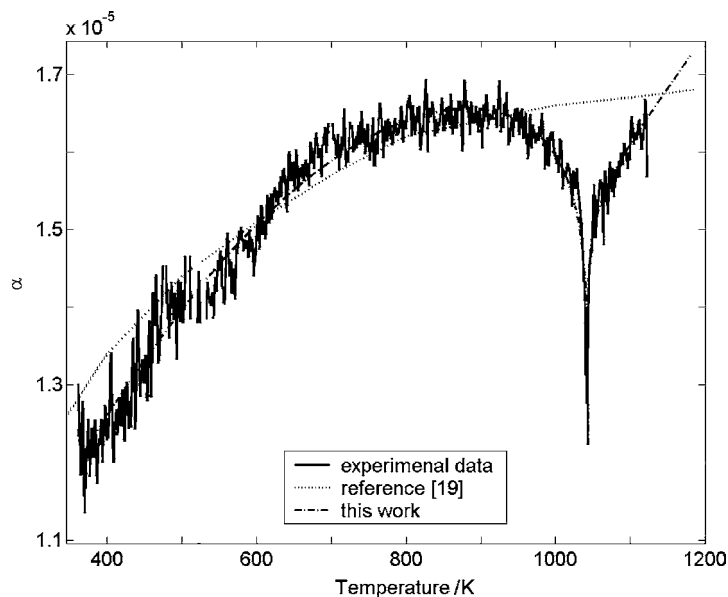


Fig. 9. Comparison of the measured (with a heating rate of 0.08 K min^{-1} in $350 \text{ K} < T < 550 \text{ K}$ and 5 K min^{-1} in $550 \text{ K} < T < 1150 \text{ K}$) and optimized linear thermal expansion coefficients (α) by Eq. (5) of ferritic Fe.

5. Conclusions

1. To accurately determine the thermal dilatation behavior of materials and the time and temperature dependence of the dilatation associated with phase transformations, it is imperative that (even) using a differential dilatometer, correction is made for subtle instrumental effects as due to minor inhomogeneities of the temperature field (leading to different displacements for both push rods) in the dilatometer and of the microstructure of the materials used for the inner dilatometer parts that are subjected to temperature change.
2. The length change observed has to be calibrated differently for heating and cooling. This calibration can be well done on the basis of a calibration measurement using a sapphire single crystal for which the thermal length change data are known.
3. The temperature calibration for both heating and cooling can be adequately performed utilizing the hysteresis-free Curie point temperature of corresponding materials.
4. On the basis of the calibration correction methods dealt with in this paper an update of the linear thermal expansion coefficient data for the ferrite and austenite phases of pure iron is possible. The linear thermal expansion of

pure iron (for the ferrite and austenite phases) was updated. For the first time the magnetic contribution to the linear thermal expansion coefficient was introduced in an analytical expression of the thermal expansion coefficient of ferritic Fe.

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