



Intercomparison of Thermophysical Property Measurements on Iron and Steels

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Abstract

The German Thermophysics Working Group (AKT) within the Society for Thermal Analysis (GEFTA) initiated and conducted an intercomparison with the objective of the determination of the thermophysical properties of pure iron, an interstitial-free steel, and a multi-phase steel. The values of heat capacity, thermal diffusivity, thermal expansion, and thermal conductivity were measured from 20 °C up to 1000 °C. In all cases, a mean value could be derived. In the case of pure iron, the mean values are in good agreement with the literature values. For the values of the thermal expansion coefficient and thermal diffusivity, the relative uncertainties are below 4 %. The relative uncertainties for the specific heat are between 4 % and 5 % up to 600 °C. Above this temperature, the uncertainty is in the range from 6 % to 8 %. The relative uncertainty of the thermal conductivity values is about 6 % below 600 °C and up to 9 % above.

Keywords IF (interstitial-free) steel · Intercomparison · Linear expansion coefficient · Multi-phase steel · Pure iron · Specific heat · Thermal conductivity · Thermal diffusivity

List of Symbols

a	Thermal diffusivity, $\text{m}^2 \cdot \text{s}^{-1}$
c_p	Specific heat, $\text{J} \cdot \text{kg}^{-1} \text{K}^{-1}$
e	Specific extinction coefficient, $\text{m}^2 \cdot \text{kg}^{-1}$
E	Normalized deviation, 1
L	Length, m
T	Temperature, K, °C
U	Uncertainty, depends
x	Measurement value, depends

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Greek symbols

α	Coefficient of thermal expansion, K^{-1}
λ	Thermal conductivity, $\text{W}\cdot\text{m}^{-1}\text{K}^{-1}$
ρ	Density, $\text{kg}\cdot\text{m}^{-3}$

Subscripts

p	Constant pressure
lab	Experimentally determined value
$mean$	Mean value
n	Normalized

Abbreviations

CIPM	Comité International des Poids et Mesures
CTE	Linear thermal expansion coefficient
DSC	Differential scanning calorimeter
GEFTA	Society for Thermal Analysis
GUM	Guide to the Expression of Uncertainty in Measurement
ICP-OES	Coupled Plasma Optical Emission Spectrometry
IF	Interstitial free
LVDT	Linear variable differential transformer
MP	Multi-phase
STA	Simultaneous thermal analysis

1 Introduction

The knowledge of the thermophysical properties of steels, e.g., thermal conductivity, heat capacity, thermal diffusivity, and thermal expansion, in a wide temperature range is of vital interest for industry [1]. Engineers need these data for quality control, safe material processing, and product engineering. Regarding to the two last points, the data were used in multi-physics simulations in a wide range of industrial applications. Unreliable data in this context can lead to material failure, increasing R&D costs, and trade distortions.

In the past, the German Thermophysics Working Group (AKT) within GEFTA initiated and conducted several intercomparisons in the field of thermophysical properties. Two intercomparisons on steel were already carried out by the AKT. In the 1980s [2] and from 2002 till 2003 [3], the thermophysical data of a high-temperature austenitic stainless steel were investigated. It should be noted that in the 1980s the derived relative uncertainties were in the range from 2 % to 2.5 % for specific heat, 2 % to 3 % for thermal conductivity values, and 8 % for thermal diffusivity. At this time, the thermal conductivity was determined by direct methods. The determination of the thermal conductivity by direct methods led to values with less uncertainties in comparison to the non-direct determined values by using the experimental values of thermal diffusivity, density, and specific heat:

$$\lambda(T) = a(T) \cdot c_p(T) \cdot \rho(T). \quad (1)$$

In 2003, the derived relative uncertainties for the mean values of the recommended thermophysical properties were 3 % for thermal expansion, 5 % for specific heat and thermal diffusivity, and 6 % to 8 % for thermal conductivity. Comparing these values with those from the former intercomparison, the relative uncertainties of the thermal conductivity values increased because in the most cases they were calculated according to Eq. 1 using the thermal diffusivity, specific heat, and density values.

Within the frame of this work technical pure iron, multi-phase (MP) steel and an interstitial-free (IF) steel were investigated. Pure iron was chosen to have the possibility to compare the results with reliable literature values [4, 5] and to get a statement about the general quality of the measurements. The MP steel and IF steel were products of the thyssenkrupp Steel Europe AG/Germany for which no literature values were available.

The participants were asked to deliver thermophysical properties at 20 °C and in the range from 100 °C to 1000 °C in steps of 100 K. The measurements should be performed in inert gas atmosphere. Further specification was not given. Participants should measure to the best of their ability according to their own laboratory standards.

The objectives of this intercomparison are to improve the measurement quality in the participating laboratories, to evaluate the applied measurement methods, and to provide reliable thermophysical data for the two investigated steels. Additionally, after completion of the intercomparison the participants are in the possession of a set of specimen with reliable values of thermophysical properties for their own use.

2 Participants and Measurement Methods

2.1 Participants

The following institutes, universities, and companies participated in this intercomparison:

- Austrian Foundry Research Institute (ÖGI), Leoben/Austria.
- Austrian Research Centers, Vienna/Austria.
- Bavarian Center for Applied Energy Research (ZAE Bayern), Würzburg/Germany.
- Fraunhofer Institute for Ceramic Technologies and Systems IKTS, Dresden/Germany.
- Fraunhofer Institute for Silicate Research ISC, Fraunhofer Center for High Temperature Materials and Design HTL, Bayreuth/Germany.
- Institute of Heat Technology and Thermodynamics (IWTT), Freiberg/Germany.
- IAB Weimar gGmbH, Weimar/Germany.
- Netzsch-Gerätebau GmbH, Selb/Germany.
- RWTH Aachen, Institute for Materials Applications in Mechanical Engineering, Aachen/Germany.
- SGL Carbon GmbH, Meitingen/Germany.

- thyssenkrupp Steel Europe AG, Duisburg/Germany.

In the following, the participants will be referred to anonymously as Lab 1 to Lab 10. Subgroups, such as Lab 1.1, Lab 1.2, identify different measurement methods of a participant.

2.2 Measurement Methods

2.2.1 Specific Heat

The majority of the participants (Lab 2, 4, 5, 6, and 9.1) used differential scanning calorimeters (DSC) by Netzsch DSC 404 x, where x denotes different modifications (x = C, F1). Additionally, DSC from TA Instruments (DSC Q100, Lab 7, and Q2000, Lab 9.2), Seteram (Multi-HTC, Lab 8), STA 449 F5 (Lab 1), STA 449 C (Lab 3), and STA 409 CD (Lab 10) from Netzsch were used. In a DSC measurement, a reference sample and the analyzed specimen are subjected to a controlled temperature program with constant heating and cooling rates. Due to the difference of heat capacities of reference material and specimen, a temperature difference between the specimen and reference material occurs at a constant heating rate, which is measured as a function of time. This temperature difference is correlated to the heat capacity of the specimen [6].

In one case, the specific heat was derived by evaluation of the laser-flash signal using Netzsch LFA Hyperflash 467 (Lab 9.3). The temperature increase of the specimen due to the adsorbed radiation of the pulse is correlated to its heat capacity. The comparison of the temperature increase for a reference material with known values of heat capacity under equal experimental conditions allows the derivation of the specific heat values of the specimen [7].

2.2.2 Thermal Diffusivity

Thermal diffusivity was determined by a standard flash method [8]. In this method, a laser pulse or light flash is absorbed at the front surface of the specimen. From the time-dependent temperature rise at the rear side of the specimen, which is monitored by an infrared detector, the thermal diffusivity is derived. Eight participants used measurement equipment by Netzsch, Germany, ($5 \times$ LFA 427, Lab 1, 2, 5, 6, and 8), $1 \times$ LFA 457 (Lab 3.1), $2 \times$ LFA 467 Hyperflash (Lab 4 and 9.2). Three participants determined the thermal diffusivity by self-built laser-flash setups (Lab 3.2, Lab 7, and Lab 9.1).

2.2.3 Linear Expansion

The linear expansion coefficient was determined by push-rod dilatometers. The participants used equipment by Netzsch DIL 402 c/ES (8 participants, Lab 1, 2, 3, 4, 5, 6, 7, and 8), Linseis L75PT (Lab 11) and Bähr DIL 802 (Lab 9).

In general, a push-rod dilatometer consists of a heated zone, containing the specimen under investigation, and an extensometer. A rod or tube made of an inert material transfers the specimens' extension out of the heated zone to the extensometer. In the measuring setups used for this report, the latter was either a LVDT or an optical linear encoder. As this method uses a mechanical transmission which is partially located in the heated zone and also contributes to the measured length change, a correction function has to be determined by means of a reference material. With the correction, the true temperature-dependent expansion $\Delta L(T)$ of the sample can be determined. The more detailed description of the measurement setup and execution is provided by Touloukian et al. [9].

The coefficient of the mean linear thermal expansion α is defined by

$$\alpha(T) = \frac{\Delta L(T)}{L_0 \cdot \Delta T}, \quad (2)$$

with $\Delta L(T) = L(T) - L_0$ and $\Delta T = T - T_0$, T : specimen temperature, T_0 : reference temperature, $L(T)$: length of the specimen at temperature T , and L_0 : length of the specimen at temperature T_0 . It should be mentioned that the coefficient of the linear thermal expansion is used to correct the dimension of a specimen as a function of the temperature, which improves the measurement precision for the determination of the thermal diffusivity and the calculation of thermal conductivity.

2.2.4 Thermal Conductivity

Thermal conductivity values were calculated according to Eq. 1 by 6 participants (Lab 1, 2, 4, 6, 8.1, and 9.1). In these cases, the experimental results of the laser-flash, specific heat, and thermal expansion measurements were used. Lab 9.2 used a Netzsch LFA 467 Hyperflash apparatus where the thermal conductivity is calculated by the implemented evaluation software. 4 participants applied also direct measurement techniques, i.e., the transient-plane-source method [10] with maximum measurement temperatures of 200 °C (Lab 3 used a TPS 500 device, Lab 8.2 used a TPS 2500S device, both from Hot Disc AB) and stationary guarded-comparative-longitudinal-heat-flow method [11] (Lab 7 and 9.3, both self-built setups). The guarded-comparative-longitudinal-heat-flow method is a stationary comparative method based on the knowledge of the thermal conductivity values of a reference material. The transient-plane-source method uses a plane heater for thermal excitation of the specimen [12]. The heater element which is embedded within the specimen consists of a meandering or spiral heating strip which deals also as temperature sensor. From the temperature increase of the sensor, the thermal conductivity is derived.

2.3 Evaluation Method

The intercomparison is aligned to the valid rules for comparison measurements, especially to the guidance laid down by the CIPM for comparison measurements. Every participant had to deliver measurement values with stated uncertainties according to GUM [13]. Mean values and the corresponding uncertainties were

calculated for all the prescribed temperatures by calculating the arithmetic mean. The normalized error function E_n was used as a criteria for the quality of a measurement value and to exclude measurements from calculating mean values. The normalized error function E_n is defined by [14]:

$$E_n = \frac{x_{lab} - x_{mean}}{\sqrt{(U_{lab}^2 + U_{mean}^2)}}, \quad (3)$$

with the measurement value x_{lab} , the calculated mean value x_{mean} , and the uncertainty of the mean value U_{mean} . An absolute value of E_n less than 1 indicates that the uncertainty stated by the laboratory concerned is reliable and the deviation of the delivered measurement value from the mean is acceptable. If the absolute value E_n was larger than 1 in any single experimental result, this point was excluded and the mean value was recalculated unless the quality criterion $-1 < E_n < 1$ was fulfilled in all results.

2.4 Description of the Sample Material

The materials investigated were technically pure iron and an interstitial-free steel as well as a multi-phase steel from thyssenkrupp Steel Europe AG. Technically pure iron with a Fe content of 99.8 % to 99.9 % is a soft and tough material, which due to its purity has a low coercive force (magnetic field strength), high magnetic saturation, and good electrical conductivity. IF steels are free of interstitially dissolved alloy contents. In this way, this steel grade receives a ferritic microstructure. They have high strength and high elongation and offer good formability. MP steel is characterized by high work hardening, which is based on a tailor-made combination of hard and soft microstructure phases. MP steel offers a tensile strength of up to 800 MPa and very good ductility in the initial state. The chemical analysis is shown in Table 1.

All specimens were prepared from the same batch of each grade of steel to ensure a high comparability of the measurement results. The available steel grades are internal reference materials of thyssenkrupp Steel Europe AG. For the homogeneity test and the stability test of the materials, there was taken a randomly chosen sample of the respective material. It was bar material where a sample at the ends and the middle section were analyzed by using inductively coupled plasma optical emission spectrometry (ICP-OES) for the chemical analysis. The results of this analysis were

Table 1 Chemical analysis of the investigated grade of the investigated samples of pure iron, IF steel, and MP steel

	C wt%	Si wt%	Mn wt%	P wt%	S wt%	Cr wt%	Mo wt%	Ni wt%	Ti wt%
Pure iron	0.001	<0.002	<0.002	<0.002	<0.001	0.001	<0.0005	<0.001	<0.0005
IF steel	0.005	0.008	0.122	0.008	0.006	0.042	0.0058	0.024	0.068
MP steel	0.156	0.67	1.9	0.009	0.002	0.32	0.0132	0.04	0.125

compared with the specification of the respective materials, and with the chemical analysis of 2005 and 2011. The ICP-OES was chosen because this method is characterized by a low limit of quantification and multi-element capability. Furthermore, this is an accredited method at thyssenkrupp Steel Europe AG.

The participants were asked for needed amount and geometry of specimens for the different measurement techniques. In most cases, three specimen for each material and measurement method were requested. The preparation of more than 420 specimens was performed by thyssenkrupp Steel Europe AG. All specimens were assembled by the process of wire erosion. Wire erosion is a shaping cutting process that works on the principle of spark erosion. After the preparation of the specimens, the eroded residues were removed in a bath of 15 % hydrochloric acid, pickling inhibitor and distilled water. Subsequently, the specimens were cleaned with ethanol in an ultrasonic bath. No heat treatment were performed after these preparation steps and nor any further heat treatment was recommended to the participants prior the measurements.

The densities at 20 °C for the investigated materials were determined by the participants (cf. Table 2).

3 Results

3.1 Specific Heat

The experimental derived mean values of heat capacity for pure iron are depicted in Fig. 1. Additionally literature values by Wallace et al. [5] were shown. Wallace et al. used a pulse heating method and investigated high-purity iron. The Curie temperature is indicated by the narrow peak at 769 °C and the α - γ phase transformation by a temperature step at 900 °C. The two curves show an excellent agreement below the Curie temperature and above the α - γ phase transformation temperature. Figure 2 shows exemplarily the values of the normalized error function E_n for the delivered specific heat value of pure iron. Only data with values $-1 \leq E_n \leq 1$ were considered for the calculation of the mean value. The relative deviations of the delivered values for pure iron and multi-phase steel from the resulting mean values are shown in Figs. 3 and 4. Besides significant systematic deviations, most results are in good agreement within an uncertainty band of 5 % to 10 %. The deviations increase for temperatures above 600 °C. From the comparison of the two graphs, it can be also concluded that systematic deviation, as can be seen in the data of Lab 7, cannot always be explained by the applied experimental setup or used experimental

Table 2 Mean values of the material densities determined by the participants of the intercomparison

	Density $\rho/\text{kg}\cdot\text{m}^{-3}$
Pure iron	7849 ± 6
IF steel	7835 ± 12
MP steel	7776 ± 13

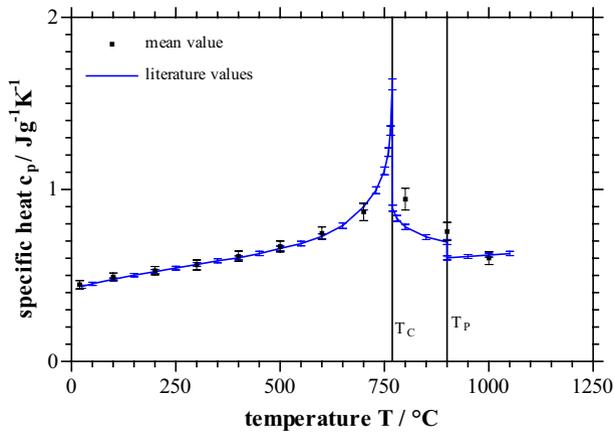


Fig. 1 Derived mean values of the specific heat of pure iron in comparison with literature values [5]. Also marked are the Curie temperature $T_C=769$ °C and the α - γ phase transformation temperature $T_P=900$ °C

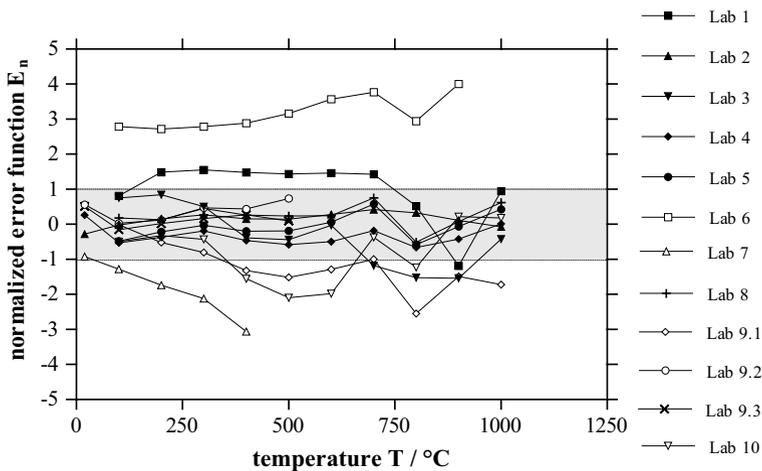


Fig. 2 Values of the normalized error function E_n (Eq. 2) for the delivered specific heat values of pure iron. Only data with values $-1 \leq E_n \leq 1$ were considered for the calculation of the mean value

procedure. In some cases, e.g., the results of laboratory 6, the results are satisfactory for MP steel (Fig. 4), but show systematic deviations for pure iron (Fig. 3).

All derived mean values for the specific heat are compiled in Table 3 and shown in Fig. 5. The relative uncertainties increase from about 5 % to 8 % at higher temperatures (cf. Fig. 13). The major contributions to the reported uncertainties were the uncertainties in the determination of the heat fluxes, the occurrence of oxidation processes of the specimens at higher temperatures due to a residual oxygen content in the inert gas atmosphere, and also uncertainties in the temperature measurement.

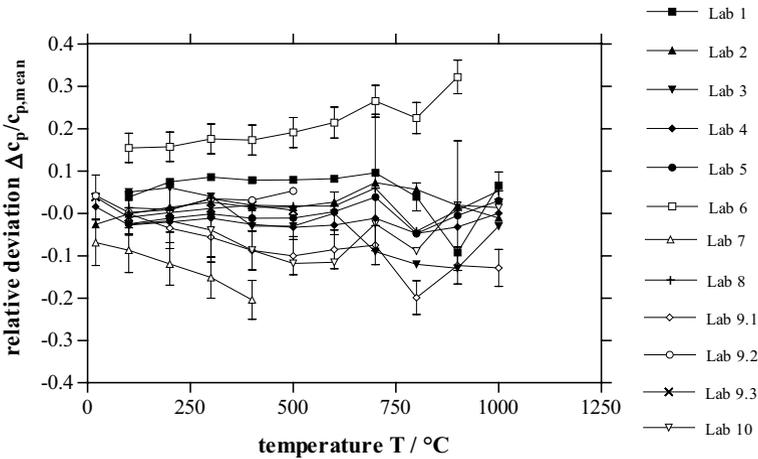


Fig. 3 Relative deviation of the specific heat values of pure iron delivered by the participants from the derived mean values. Only few uncertainty bars were plotted in order to keep the overview

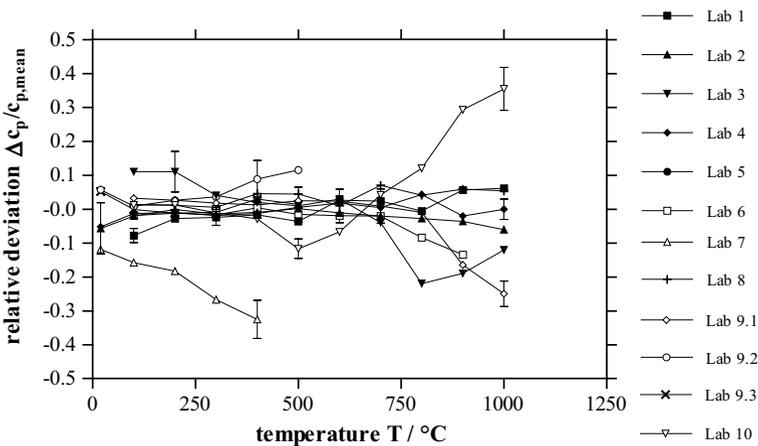


Fig. 4 Relative deviation of the specific heat values of MP steel delivered by the participants from the derived mean values. Only few uncertainty bars were plotted in order to keep the overview

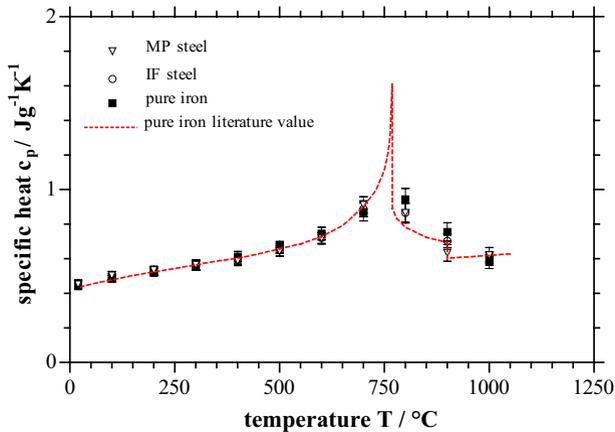
Typical relative mass losses during the measurements were in the low single-digit per mill range.

3.2 Thermal Diffusivity

In Fig. 6, the calculated mean values of the thermal diffusivity for investigated materials are depicted as a function of temperature (cf. Table 4). The values for pure iron are compared with recommended literature values [15] and an excellent agreement can be stated within the given uncertainties. The thermal diffusivity values of IF

Table 3 Derived mean values of the specific heat of pure iron, IF steel, and MP steel

Temperature T/°C	Specific heat of pure iron $c_p/\text{Jg}^{-1}\text{K}^{-1}$	Specific heat of IF steel $c_p/\text{Jg}^{-1}\text{K}^{-1}$	Specific heat of MP steel $c_p/\text{Jg}^{-1}\text{K}^{-1}$
20	0.447 ± 0.024	0.454 ± 0.024	0.452 ± 0.024
100	0.493 ± 0.021	0.484 ± 0.020	0.504 ± 0.023
200	0.529 ± 0.024	0.522 ± 0.022	0.534 ± 0.023
300	0.562 ± 0.029	0.560 ± 0.027	0.566 ± 0.028
400	0.613 ± 0.030	0.596 ± 0.027	0.587 ± 0.026
500	0.669 ± 0.032	0.661 ± 0.030	0.645 ± 0.030
600	0.746 ± 0.036	0.721 ± 0.032	0.718 ± 0.033
700	0.871 ± 0.051	0.890 ± 0.046	0.912 ± 0.048
800	0.944 ± 0.063	0.866 ± 0.054	0.866 ± 0.058
900	0.756 ± 0.053	0.705 ± 0.041	0.634 ± 0.048
1000	0.602 ± 0.037	0.585 ± 0.040	0.620 ± 0.045

**Fig. 5** Derived mean values of the specific heat of pure iron and the two investigated steels in comparison with literature values for pure iron [5]

steel are in the same order of magnitude as of iron with a smaller increase towards to 20 °C. Significant smaller values for temperature below T_c were found for the MP steel. At 20 °C, a thermal diffusivity of $(8.84 \pm 0.30) \text{ mm}^2 \cdot \text{s}^{-1}$ was determined. All materials show a minimum of the thermal diffusivity at the Curie temperature.

The major contributions to the reported uncertainties in Table 4 were the uncertainties in the determination of the specimen thickness at the measurement temperature and in the use of the proper evaluation model. Also the determination of the measurement temperature is critical. At lower temperatures, the thermal diffusivity values depend strongly on the temperature in the case of pure iron and IF steel. At higher, the determination of the specimen temperature is affected with higher uncertainties.

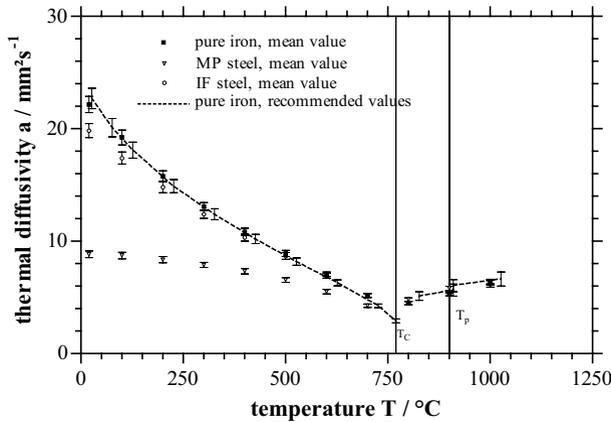


Fig. 6 Determined thermal diffusivity values of the investigated metals as a function of temperature. Additionally recommended values for pure iron [15] and specific temperatures for iron (Curie temperature T_c and α - γ phase transformation temperature T_p) are depicted

Table 4 Derived mean values of the thermal diffusivity of pure iron, IF steel, and MP steel

Temperature T/°C	Thermal diffusivity of pure iron $a/mm^2 \cdot s^{-1}$	Thermal diffusivity of IF steel $a/mm^2 \cdot s^{-1}$	Thermal diffusivity of MP steel $a/mm^2 \cdot s^{-1}$
20	22.16 ± 0.72	19.84 ± 0.63	8.84 ± 0.30
100	19.23 ± 0.67	17.40 ± 0.53	8.73 ± 0.29
200	15.78 ± 0.48	14.82 ± 0.50	8.36 ± 0.28
300	13.08 ± 0.37	12.41 ± 0.38	7.89 ± 0.23
400	10.84 ± 0.31	10.33 ± 0.31	7.33 ± 0.24
500	8.91 ± 0.27	8.69 ± 0.30	6.55 ± 0.21
600	7.06 ± 0.19	6.92 ± 0.22	5.51 ± 0.20
700	5.18 ± 0.18	5.16 ± 0.17	4.26 ± 0.16
800	4.49 ± 0.15	4.50 ± 0.17	4.82 ± 0.15
900	5.27 ± 0.14	5.29 ± 0.16	5.80 ± 0.19
1000	6.33 ± 0.23	6.39 ± 0.22	6.07 ± 0.15

Exemplary, the deviation of the delivered thermal diffusivity values from the resulting mean value for the MP steel is shown in Fig. 7. Values determined by self-build setups are depicted with red symbols. Below 600 °C, the relative deviations are smaller than 5 %. Above 600 °C, the deviations increase up to 13 %.

3.3 Thermal Expansion

Exemplary, the experimentally derived values of the linear thermal expansion $\Delta L/L_0$ for each of the three materials is shown in Fig. 8 (data set Lab 9). In case of iron, the phase transition at about 900 °C (bcc/fcc) results in a sharp bend shrinkage of the

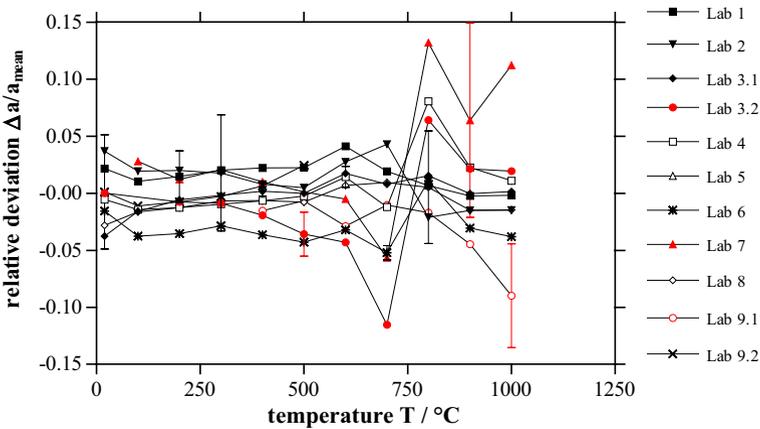


Fig. 7 Relative deviation of thermal diffusivity values of MP steel delivered by the participants from the derived mean values. Red marked symbols indicates results from self-build setups. The lines are only guides to the eyes

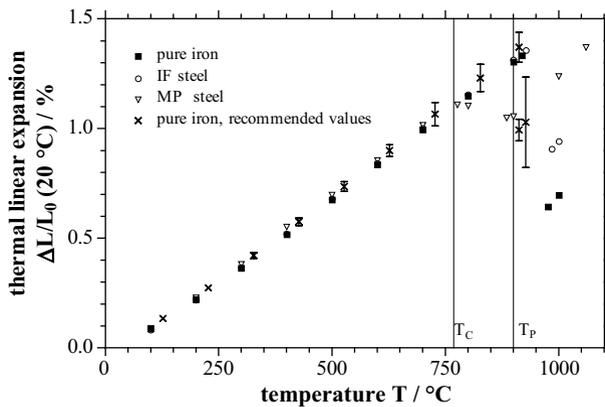


Fig. 8 Experimentally determined linear thermal expansion $\Delta L/L_0$ versus temperature for pure iron, IF steel, and MP steel in comparison to the recommended values for pure iron [9]. The data were delivered by Lab 9

specimen. The values for the linear thermal expansion coefficient α were derived from the relative length change according to Eq. 2. The reference temperature T_0 was 20 °C. At this temperature, the reference length L_0 was determined and also ΔT was calculated. The resulting mean values for all three material of all participants are shown in Fig. 9.

Table 5 shows a compilation of all derived mean values for the linear thermal expansion coefficient of all investigated materials. The major contribution to the reported uncertainties lies in the determination of the reference length at a defined temperature. The influence of this value is especially important at low

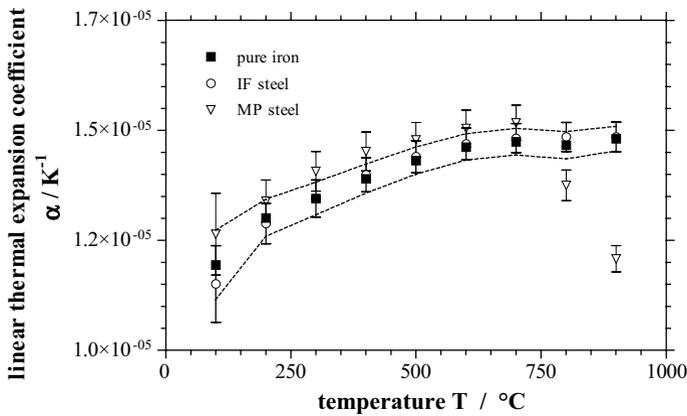


Fig. 9 Linear thermal expansion coefficient as a function of temperature for all investigated three metals. The dashed lines indicate the uncertainty interval for the pure iron data. The reference temperature T_0 was 20 °C

Table 5 Derived mean values of the linear thermal expansion coefficient of pure iron, IF steel, and MP steel

Temperature T/°C	CTE of pure iron $\alpha/10^{-5} K^{-1}$	CTE of IF steel $\alpha/10^{-5} K^{-1}$	CTE of MP steel $\alpha/10^{-5} K^{-1}$
100	1.194 ± 0.079	1.151 ± 0.087	1.264 ± 0.093
200	1.301 ± 0.042	1.288 ± 0.046	1.339 ± 0.048
300	1.345 ± 0.037	1.345 ± 0.042	1.407 ± 0.045
400	1.390 ± 0.033	1.399 ± 0.039	1.452 ± 0.044
500	1.431 ± 0.031	1.440 ± 0.036	1.478 ± 0.040
600	1.462 ± 0.030	1.469 ± 0.036	1.504 ± 0.042
700	1.474 ± 0.031	1.482 ± 0.033	1.517 ± 0.040
800	1.466 ± 0.031	1.485 ± 0.033	1.375 ± 0.035
900	1.481 ± 0.028	1.485 ± 0.034	1.208 ± 0.030

temperatures. At higher temperatures, the influence decreases. Usually, the most critical parameter is the determination of the correct specimen temperature. As the measurement is non-stationary, the specimen is not homogeneously heated and the average temperature follows the temperature of the surrounding. Thus, an adequate heating rate has to be chosen. For the determination of the correction function, a reference material of the same size as the specimen but known expansion behavior has to be used. The choice of a suitable reference material (no recrystallization within the temperature range) and its dimensions (as equal as possible to the specimens' dimensions) is crucial.

For an assessment of the uncertainties, the relative deviation $\Delta\alpha/\alpha_{mean}$ was calculated and is shown exemplarily for IF steel in Fig. 10. Also shown (in gray) are

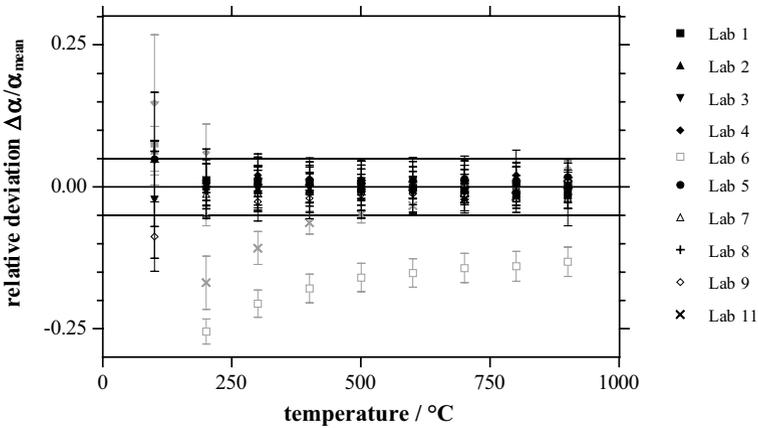


Fig. 10 IF steel, relative deviation of the CTE from the mean value as a function of the temperature. Data points indicated by gray symbols are not used for calculation of the mean value

the deviations of corresponding values which are considered to be not reliable according to Eq. 3.

3.4 Thermal Conductivity

The estimated mean values of thermal conductivity for all investigated metals are depicted in Fig. 11 and compiled in Table 6. Comparing the values for iron with recommended literature values [4], a small systematic deviation of +3 % can be observed. The thermal conductivity of IF steel and iron are similar, although the thermal conductivity of IF steel is systematically lower. The thermal conductivity of

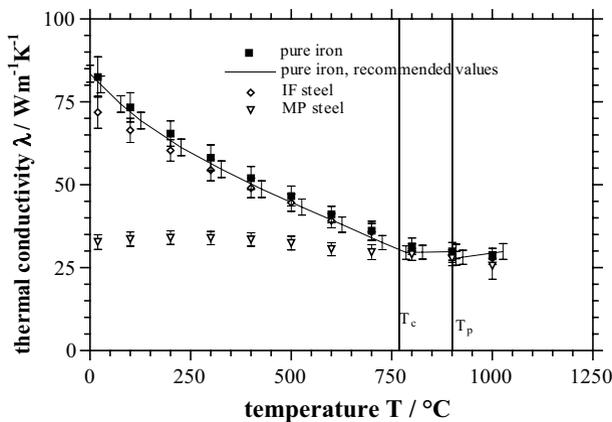


Fig. 11 Determined thermal conductivity values for all investigated metals as a function of temperature. For pure iron also, the recommended literature values [4] and specific temperatures (Curie temperature T_c and α - γ phase transformation temperature T_p) are shown

Table 6 Derived mean values of the thermal conductivity of pure iron, IF steel, and MP steel

Temperature T/ °C	Thermal conductivity of pure iron $\lambda/W\cdot m^{-1}K^{-1}$	Thermal conductivity of IF steel $\lambda/W\cdot m^{-1}K^{-1}$	Thermal conductivity of MP steel $\lambda/W\cdot m^{-1}K^{-1}$
20	82.5 ± 6.1	71.9 ± 5.3	32.8 ± 2.4
100	73.4 ± 4.4	66.4 ± 3.6	33.6 ± 2.1
200	65.5 ± 3.9	60.4 ± 3.2	34.1 ± 2.0
300	58.2 ± 3.8	54.4 ± 3.2	34.0 ± 2.0
400	52.0 ± 3.5	49.1 ± 2.9	33.5 ± 2.0
500	46.7 ± 3.0	44.6 ± 2.6	32.5 ± 2.1
600	41.1 ± 2.5	39.2 ± 2.2	30.6 ± 1.9
700	36.2 ± 2.8	35.8 ± 2.5	29.7 ± 2.2
800	31.5 ± 2.5	29.7 ± 2.5	29.0 ± 1.9
900	30.0 ± 2.7	28.6 ± 1.9	27.9 ± 2.2
1000	28.4 ± 2.5	27.9 ± 1.9	25.6 ± 4.1

the multi-phase steel is only weakly dependent from temperature in the investigated temperature range (cf. Fig. 11).

Figure 12 shows the relative deviation of all delivered thermal conductivity values from the calculated mean value. Below 700 °C most curves show a deviation less than 10 %. The red data points indicate the measurement values derived by self-build setups.

The reported uncertainties in Table 6 result mainly from the application of the law of error propagation on Eq. 1. As already mentioned, only 4 participants used direct methods and only at temperatures below 400 °C. Therefore, the uncertainties of the mean values are not much affected by these direct methods. In the case of

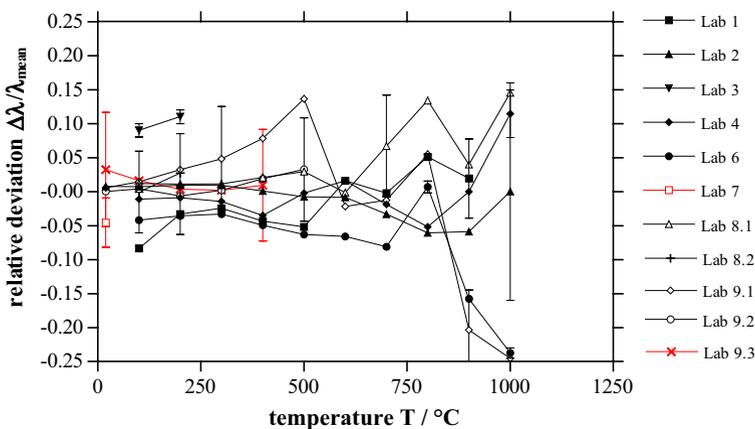


Fig. 12 Relative deviation of the thermal conductivity values of MP steel, delivered by the participants, from the derived mean values. Red marked symbols indicates results from self-build setups. The lines are only guides to the eyes

the transient-plane-source method, thermal contact resistances between sensor and specimen could lead to higher uncertainties. With the guarded-comparative-longitudinal-heat-flow method, the correct installation of the temperature sensors at the specimen and reference samples at defined locations and with a good thermal contact is necessary to avoid higher uncertainties.

4 Discussion

Although the focus of this intercomparison was the determination of the thermophysical properties of the IF steel and MP steel, the additional consideration of pure iron, whose properties are well known, was essential to assess the reliability of the derived data. In this way, it is possible to separate effects resulting from the different experimental procedures from the real properties of the investigated steels. For pure iron, the agreement of the derived mean values with recommended literature values is generally excellent. Just above the Curie temperature, where a sharp drop occurs, the specific heat data of this work were systematically higher than the literature values [5]. This can be explained by the different dynamics of the experimental methods used by Wallace et al. and the participants and the fact that the specimens within the DSC measurements were not in the state of thermal equilibrium. Where in the DSC measurements typical heating rates between $10 \text{ K}\cdot\text{min}^{-1}$ and $20 \text{ K}\cdot\text{min}^{-1}$ were applied and the specimen is not homogeneously heated up, the pulse heating calorimetry operates with heating rates between $10^3 \text{ }^\circ\text{C}\cdot\text{s}^{-1}$ and $10^4 \text{ }^\circ\text{C}\cdot\text{s}^{-1}$ and homogeneous volume heating. Therefore, it can be concluded that the general approach of the intercomparison will deliver reasonable results for the thermophysical properties of the IF steel and MP steel.

Besides a few exceptions, the variation of the determined values for the CTE is comparatively small, though, as one can see from Fig. 10, significant deviations particularly occur at low temperatures. Most likely this is due to the uncertainty in the determination of the reference length which should be performed with a high precision. Other potential uncertainties arise, i.e., from the determination of the true specimen temperature or the uncertainty of the reference material used for correction.

Discussing the relative deviations from the calculated mean values, it is obvious that above $600 \text{ }^\circ\text{C}$ the deviations significantly increase concerning the specific heat and thermal diffusivity measurements (cf. Figs. 3, 4, and 7). Above $600 \text{ }^\circ\text{C}$, the influence of the ferromagnetic/paramagnetic transition at the Curie temperature is visible. In this temperature range, the results of laser-flash measurements are more sensitive from the applied pulse energies. In case of specific heat measurements, starting oxidation processes at higher temperatures could lead to deviations. For all methods, a reliable temperature calibration is important to reduce the uncertainties.

In Fig. 13, the resulting uncertainties for the derived mean values of the thermophysical properties as a function of the temperature are depicted. For the values of the thermal expansion coefficient and thermal diffusivity, the relative uncertainties are below 4 %. The relative uncertainties for the specific heat are between 4 % and 5 % up to $600 \text{ }^\circ\text{C}$. Above this temperature, the values increase for all materials significantly in the range of 6 % to 8 %. According to the law of error propagation,

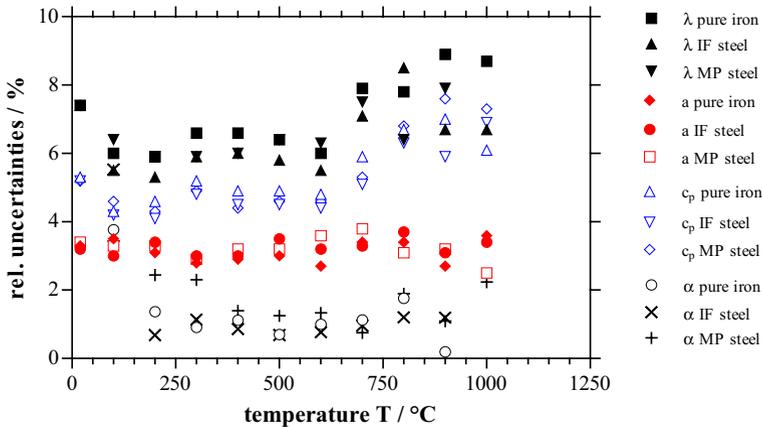


Fig. 13 Derived relative uncertainties for the determined mean values for the investigated materials as a function of temperature

the relative uncertainties for the thermal conductivity values increases to about 6 % below 600 °C and up to 9 % above.

The comparison of the results of this approach with previous intercomparisons on steels in the 1980s, earlier mentioned, clearly shows the progress in measurement techniques. Especially, the laser-flash technology was improved by advanced electronics and evaluation methods and average uncertainties decreases from about 8 % to 3.5 %. On the other hand, the average uncertainties of the specific heat measurements are higher than in the past (1980 about 2.5 %, 2003 about 5 %). Since the majority of the thermal conductivity values were calculated according to Eq. 1, the corresponding uncertainty values increase significantly at temperatures above 600 °C.

5 Conclusion

The following main conclusions can be drawn from the results of this intercomparison:

The average values of the thermophysical properties of the investigated metals can be determined with a sufficient low uncertainty within the frame of this intercomparison.

In some cases, despite the statement of low uncertainties of some participants, the delivered measurement values show significant deviations from the mean value outside the acceptable range. This demonstrates that the performance of intercomparisons is important to ensure the high-quality standard of laboratories in the daily routine.

The indirect determination of the thermal conductivity by the use of thermal diffusivity, specific heat, and thermal expansion values leads to a high uncertainty of the thermal conductivity values. It is trivial, but also necessary, to determine all

quantities necessary for the calculation of thermal conductivity with sufficiently small uncertainties. In this case, the relatively high uncertainty values for the specific heat, especially above 600 °C, have a negative influence. In this context, it is desirable to implement more and diverse thermal conductivity measuring methods in similar intercomparisons in the future. Furthermore, it is important to provide methods that allow the direct determination of the thermal conductivity of metals with a low uncertainty, especially at high temperatures.

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References

1. J. Bojkovski et al., A roadmap for thermal metrology. *Int. J. Thermophys.* **30**, 8 (2009)
2. L. Binkele, *Austenitic Chromium Nickel Steel as Standard Reference Material in Measurement of Thermal and Temperature Conductivity* (Butterworth-Heinemann, Waltham, 1990)
3. S. Rudtsch et al., Intercomparison of thermophysical property measurements on an austenitic stainless steel. *Int. J. Thermophys.* **26**, 855–867 (2005)
4. Y.S. Touloukian et al., *Thermal Conductivity: Metallic Elements and Alloys. Thermophysical Properties of Matter, v. 1* (IFI/Plenum, New York, 1970)
5. D.C. Wallace, P.H. Sidles, G.G. Danielson, Specific heat of high purity iron by a pulse heating method. *J. Appl. Phys.* **31**, 8 (1960)
6. I. Maglic, A. Cezairliyan, V.E. Peletsky, *Compendium of Thermophysical Property Measurement Methods: Volume 2 Recommended Measurement Techniques and Practices* (Plenum Press, New York, 1992)
7. K. Shinzato, T. Baba, A laser flash apparatus for thermal diffusivity and specific heat capacity measurements. *J. Therm. Anal. Calorim.* **64**, 413–422 (2001)
8. W.J. Parker et al., Flash method of determining thermal diffusivity, heat capacity, and thermal conductivity. *J. Appl. Phys.* **32**, 1679–1684 (1961)
9. Y.S.K. Touloukian, R.K. Taylor, R.E. Desai, *Thermal Expansion Metallic Elements and Alloys*, vol. 12, Thermophysical Properties of Matter—the TPRC Data Series (University microfilms international, Ann Arbor, 1975)
10. S.E. Gustafsson, Transient plane source techniques for thermal conductivity and thermal diffusivity measurements of solid materials. *Rev. Sci. Instrum.* **62**, 797–804 (1991)
11. ASTM, *E1225-13, Standard Test Method for Thermal Conductivity of Solids Using the Guarded Comparative-Longitudinal Heat Flow Technique*, ASTM International, West Conshohocken, PA, 2013 (ASTM International, West Conshohocken, 2013)
12. ISO, *22007-2:2015 Plastics—Determination of Thermal Conductivity and Thermal Diffusivity—Part 2: Transient Plane Heat Source (Hot Disc) Method* (International Organization for Standardization, Geneva, 2015)
13. GUM, *Evaluation of Measurement Data—Guide to the Expression of Uncertainty in Measurement* (International Organization for Standardization, Geneva, 1995)
14. EAL-P7, *EAL Interlaboratory Comparisons*. 1996. Ed. 1
15. Y.S. Touloukian et al., *Thermophysical Properties of Matter: Thermal Diffusivity* (IFI/Plenum, New York, 1974)

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