

## Intercomparison of Thermophysical Property Measurements on an Austenitic Stainless Steel

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The results of an inter laboratory comparison of thermal conductivity, thermal diffusivity, specific heat capacity, and thermal expansion measurements on austenitic stainless steel in the temperature range between 20 and 1000°C are presented here. Mean values are presented for the physical properties studied. Reliable relative expanded uncertainties can be stated for the properties determined, which were achieved by applying good measurement practice, i.e., 3% for thermal expansion, 5% for specific heat capacity and thermal diffusivity, and 6% for thermal conductivity. The mean values derived from this intercomparison agree well with the results of a previous intercomparison in 1990.

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**KEY WORDS:** austenitic stainless steel; specific heat capacity; thermal conductivity; thermal diffusivity; thermal expansion.

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## 1. INTRODUCTION

Many industrial applications necessitate the determination of accurate thermophysical properties, especially at high temperatures. A large number of instruments for this purpose have been developed by research institutes or are already commercially available. Well-trained and experienced personnel as well as reference materials are required to ensure reliable results and to check the credibility of the uncertainty budget. Even though the *ISO Guide to the Expression of Uncertainty in Measurement* [1] is generally accepted for the evaluation of uncertainty, its application is a very complex procedure with many degrees of freedom with respect to the used model and the description of the influence of different contributions. As a result, uncertainty budgets for thermophysical property measurements often considerably deviate from each other, even if the same type of instrument is used. Unfortunately, few reference materials are available for checking the uncertainty budget of thermophysical property measurements at high temperatures.

During the 1980s, the German Thermophysics Working Group characterized an austenitic stainless steel as a reference material for thermophysical properties [2]. The same material was used in this intercomparison by members of that working group. The following thermophysical properties were measured: linear thermal expansion, thermal conductivity, thermal diffusivity, and specific heat capacity.

The aim of the intercomparison was to provide a high-temperature standard for the participating laboratories and to check the credibility of the uncertainty budgets. Furthermore, comparing the current results with the previous ones from 1990 will establish whether modern measuring equipment and improved evaluation software deliver better results.

## 2. PROCEDURE DETAILS

### 2.1. Material Characterization and Preparation

The material investigated was a high-temperature austenitic stainless steel X 10 NiCrMoTiB 15 15 (Material No. 1.4970). All samples were prepared from the same batch of material which was stored at the Forschungszentrum Jülich (Jülich Research Centre). CrNi steel is attractive due to its high thermal strength values and recrystallization temperature. An analysis of the constituents is shown in Table I. Further details can be found in Ref. 2. The density of the material at 20°C is  $\rho = (7970 \pm 15) \text{ kg}\cdot\text{m}^{-3}$ . All samples were annealed in argon after preparation for 30 min at 1050°C and subsequently quenched in a distilled water bath.

**Table I.** Chemical Composition of the Investigated Steel X 10 NiCrMoTiB 15 15 According to DIN Specifications (Fe remaining percentage)

Element	C	Si	Mn	P	S	Cr	Ni	Mo	Ti
(Mass%)	0.08–0.012	0.25–0.45	1.60–2.00	≤0.03	≤0.015	14.5–15.5	15.0–16.0	1.05–1.25	0.35–0.55

## 2.2. Participants

Measurements were carried out by universities, R&D institutes, and national metrological institutes (Table II) from two countries: ARC Seibersdorf Research GmbH (ARCS, Austria), Bavarian Center for Applied Energy Research (ZAE, Germany), Fraunhofer IKTS (Germany), Freiberg University of Mining and Technology (Germany), Graz University of Technology (Austria), University of Stuttgart (IKE, Germany), Österreichisches Gießerei-Institut Leoben (ÖGI, Austria), Physikalisch-Technische Bundesanstalt (PTB, Germany), Research Center Karlsruhe (FZK, Germany), and RWTH Aachen University (IKKM, Germany). The participating members are referred to anonymously in the following text as L1–L10.

## 3. MEASUREMENTS

### 3.1. Measurement Methods

#### 3.1.1. Thermal Expansion

The linear differential thermal expansion of the sample material was determined by the members L3, L5, L6, and L8 with commercial push-rod

**Table II.** Participating Laboratories and Measurement Matrix

Laboratory	Thermal expansion	Specific heat	Thermal diffusivity	Thermal conductivity
ARCS	×	×	×	×
Freiberg	×	×	×	×
FZK			×	
Graz		×		
IKE			×	×
IKKM		×	×	×
IKTS	×	×	×	×
ÖGI	×	×	×	×
PTB		×		
ZAE	×	×	×	×

**Table III.** Prepared Sample Sizes for the Dilatometer Measurements

Laboratory	Diameter (mm)	Length (mm)
L3	3 × 3 (quadratic)	20
L4	6	35
L5	6	50
L6	4	12
L8	3	50

dilatometers and thermomechanical analysis [3]. The sample sizes used are compiled in Table III.

The primary reason for doing these measurements was the need for a correction of the sample thickness at high temperatures due to its thermal expansion. It is difficult to determine sample thickness directly during thermal-conductivity or thermal-diffusivity measurements above room temperature. Therefore, the results of the thermal-expansion measurements were used to correct the sample thickness of thermal-conductivity/diffusivity measurements.

All participants were asked to deliver the temperature-dependent linear thermal expansion related to the length at 20°C stated by

$$\frac{\Delta L}{L_0} = \frac{L(T) - L_0}{L_0}, \quad (1)$$

where  $L(T)$  stands for the actual sample length at a given temperature  $T$  and  $L_0$  for the reference length at  $T = 20^\circ\text{C}$ .

### 3.1.2. Specific Heat Capacity

The specific heat capacity was determined by differential scanning calorimetry (DSC) [3]. All participants used commercial measuring equipment. Cylindrical samples with a diameter between 5 and 6 mm and a height between 0.5 and 1 mm were used for the measurements. One participant used a Calvet-type DSC with a sample length of about 16 mm.

### 3.1.3. Thermal Diffusivity

The laser-flash method [3] and the modulated light beam method [4] were used for the thermal-diffusivity measurements. Commercial as well as experimental apparatus were used. The dimensions of the used samples are given in Table IV.

**Table IV.** Prepared Sample Sizes for the Laser-Flash Measurements

Laboratory	Diameter (mm)	Thickness (mm)
L1	12.7	2–3
L3	12.7	1–2
L4	12.6	2–3
L5	12.7	3
L6	10.0	1
L8	12.5	1
L10	12.7	1

### 3.1.4. Thermal Conductivity

Commercial apparatus [3] were used to carry out the direct thermal-conductivity measurements by means of the comparative method. Furthermore, thermal-conductivity values  $\lambda$  were calculated using the results of density  $\rho$ , thermal expansion  $\Delta L/L_0$ , thermal diffusivity  $a$ , and specific heat capacity  $c_p$  measurements according to

$$\lambda(T) = a(T)\rho(T)c_p(T). \quad (2)$$

### 3.2. Measurement Uncertainties

The measurement uncertainties of all participants were determined according to the GUM [1]. Typical relative measurement uncertainties stated for the methods used are as follows: thermal-expansion measurements between 2 and 3%, thermal-diffusivity measurements 5%, direct thermal-conductivity measurements between 6 and 8%, and specific-heat-capacity measurements between 1.5 and 5%. The assigned uncertainties are expanded and the coverage factor ( $k=2$ ) corresponds to a coverage probability of approximately 95%.

### 3.3. Evaluation Methods and Averaging

All participants were asked to condense their results to one set of data for each thermophysical property and as a function of prescribed temperatures. Data are therefore available at 20°C and in steps of 50°C from 50 to 1000°C. The only instructions given about calibrating the instruments and evaluating the data were that these should be carried out with the highest degree of accuracy possible according to the state of the art and that the specifics of the instruments should be taken into consideration (good measurement practice). Reference values of the thermophysical properties were

**Table V.** Reference Values of the Intercomparison

$T$ (°C)	$\Delta L/L_0$ (%)	$c_p$ ( $J \cdot g^{-1} \cdot K^{-1}$ )	$a$ ( $mm^2 \cdot s^{-1}$ )	$\lambda$ ( $W \cdot m^{-1} \cdot K^{-1}$ )
20	0.000	0.4840	3.497	13.49
50	0.051	0.4861	3.584	13.86
100	0.132	0.5039	3.718	14.87
150	0.217	0.5153	3.834	15.64
200	0.304	0.5241	3.951	16.35
250	0.391	0.5314	4.103	17.17
300	0.487	0.5388	4.224	17.87
350	0.583	0.5461	4.346	18.59
400	0.682	0.5525	4.467	19.27
450	0.780	0.5590	4.625	20.13
500	0.879	0.5647	4.732	20.75
550	0.979	0.5703	4.891	21.59
600	1.082	0.5818	4.996	22.43
650	1.184	0.5899	5.099	23.14
700	1.286	0.5959	5.153	23.56
750	1.388	0.6019	5.248	24.16
800	1.489	0.6074	5.339	24.73
850	1.592	0.6141	5.430	25.35
900	1.695	0.6179	5.519	25.85
950	1.799	0.6232	5.615	26.44
1000	1.902	0.6295	5.709	27.07

calculated for all the prescribed temperatures by calculating the arithmetic mean (Table V). The uncertainties of the reference values were calculated according to the GUM by using the assumption of correlated results. This is due to the fact that in many cases, instruments of the same type from the same manufacturer have been used which were calibrated by means of the same reference materials. The deviation function  $E_n$  was introduced to compare the experimental results and for further discussion. The deviation  $E_n$ , normalized with respect to the experimental uncertainty  $U_{lab}$  stated by the participants, was calculated using

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

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. If the 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  $E_n < 1$  was fulfilled in all results.

## 4. RESULTS AND DISCUSSION

### 4.1. Thermal Expansion

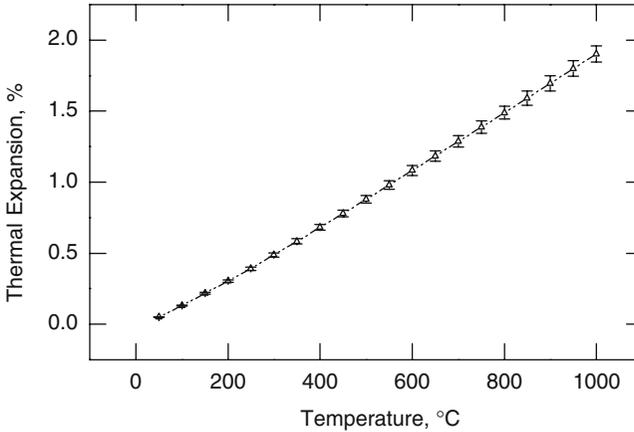
The mean values of the thermal expansion  $\Delta L/L_0$  are shown in Fig. 1. A thermal expansion of about 1.9% at 1000°C was observed. The increase in the thermal expansion is nearly linear with temperature.

The normalized deviation  $E_n$  values for thermal expansion as a function of temperature using the resulting mean value are shown in Fig. 2. Measurements of the thermal expansion were not carried out in the first intercomparison during the 1980s; data of Richter [5], a member of that working group, were used.  $E_n$ -values for these previous expansion data are also plotted as a means of comparison. It can be seen that the results of the present intercomparison are in agreement with the earlier results. The expanded uncertainties of the values given by the participants, and that of the reference value, are less than 3%. Figure 2 shows that these uncertainties are realistic.

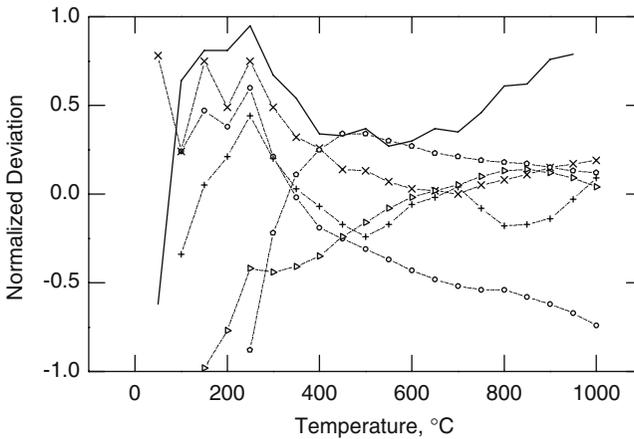
### 4.2. Specific Heat Capacity

The mean values for the specific heat capacity derived from the results of seven participants are shown in Fig. 3. A typical increase in the specific heat capacity with temperature can be observed.

The normalized deviation  $E_n$  for the specific heat capacity from the resulting mean value as a function of temperature is shown in Fig. 4. The regularly distributed deviation values indicate the high quality the specific-heat-capacity measurements of all participants. At low temperatures, i.e., at the beginning of the measurement, in some cases higher deviations were found. It is assumed that these were caused by the decrease of the sensitivity of the used high-temperature calorimeters in the low-temperature range. In comparison to the results of the previous intercomparison [2], the specific heat capacity had lower values, especially at higher temperatures (about 3% at 1000°C). Since there is no indication that the material might have changed with time and the same sample preparation procedure was used, it would appear that this significant systematic deviation is due to the methods used. The values given in the previous intercomparison were calculated as weighted mean values from the results of seven institutes using DSC, pulse calorimetry, and adiabatic calorimetry at temperatures of 600°C and below. At temperatures above 600°C, only the results of pulse calorimetry were used. The  $c_p$ -values of the present study were determined by means of DSC. It should be considered here that austenitic stainless steel is not in thermodynamic equilibrium at low temperatures as a result of being quenched in the water bath during sample preparation. At high



**Fig. 1.** Values derived for the non-weighted mean of the linear normalized thermal expansion as a function of temperature.



**Fig. 2.** Normalized deviation of the participating laboratories' results from the mean value derived as a function of temperature. Also shown is the normalized deviation of literature values [5].

temperatures the material will relax to thermodynamic equilibrium. As a result, the specific heat capacity measured depends on the thermal history of the sample, e.g., on the heating rate – which is very different for pulse calorimetry and DSC. This is supported by the observations of most participants that there was a systematic difference between the first and second runs of a specific-heat-capacity experiment carried out with the same specimen. These differences were very close to the repeatability of the DSC

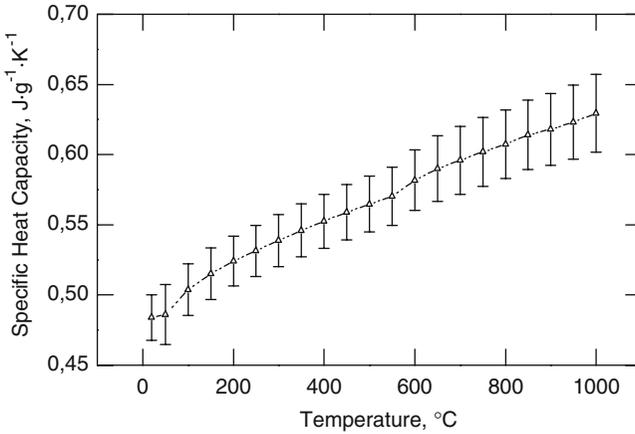


Fig. 3. Values derived for the non-weighted mean of the specific heat capacity as a function of temperature.

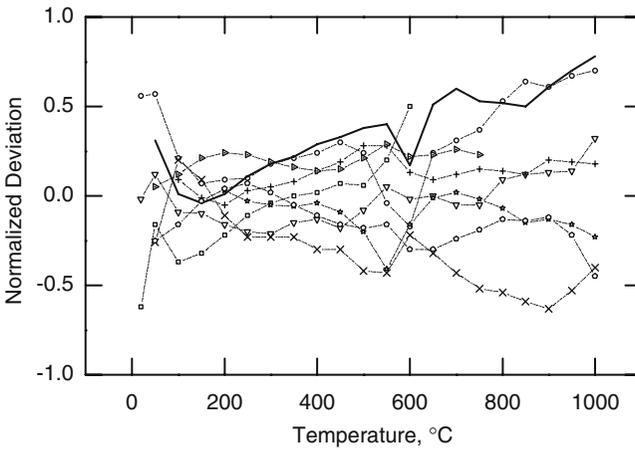


Fig. 4. Normalized deviation of the participating laboratories' specific heat capacity results and of the previous intercomparison test [2] from the mean value derived as a function of temperature.

measurements and considerably less than the stated uncertainties. The relative expanded uncertainty of the reference values is about 5% over the total temperature interval. At about 600°C a small step occurs which is interpreted as an order-disorder transition [6].

### 4.3. Thermal Diffusivity

The mean values of the thermal diffusivity derived as a function of temperature are shown in Fig. 5. The thermal diffusivity increases nearly linearly from about  $(3.50 \pm 0.18) \text{ mm}^2 \cdot \text{s}^{-1}$  at  $20^\circ\text{C}$  to  $(5.71 \pm 0.26) \text{ mm}^2 \cdot \text{s}^{-1}$  at  $1000^\circ\text{C}$ . The average relative expanded uncertainty is about 5% within the temperature range investigated.

The normalized deviation  $E_n$  concerning the thermal diffusivity is plotted in Fig. 6. The regularly distributed curves covering the range between  $-1$  and  $+1$  indicate that a relative expanded uncertainty of 5% as stated by the participants is realistic for laser-flash experiments in that temperature range.

### 4.4. Thermal Conductivity

Typical uncertainties of the direct thermal-conductivity measurements by means of the comparative method are between 6% at  $20^\circ\text{C}$  and 8% at  $1000^\circ\text{C}$ . The reference values of the thermal conductivity calculated from the reference values of the specific heat capacity and thermal diffusivity as well as from the given temperature-dependent density increase nearly linearly with temperature (see Fig. 7) from  $(13.49 \pm 0.82) \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$  at  $20^\circ\text{C}$  to  $(27.07 \pm 1.74) \text{ W} \cdot \text{m}^{-1} \cdot \text{K}^{-1}$  at  $1000^\circ\text{C}$ . The relative expanded uncertainty of the reference values is about 6% within the temperature range investigated.

The normalized deviation  $E_n$  concerning the thermal conductivity is shown in Fig. 8. The regularly distributed curves covered the range from  $-1$  to  $+1$ . The results of the direct method applied by laboratory L7 are also shown for comparison – even the normalized deviation  $E_n$  is larger than 1 for temperatures above  $200^\circ\text{C}$ . As a consequence, these data were not considered for the calculation of the reference value.

## 5. CONCLUSIONS

The results of this intercomparison on austenitic stainless steel X 10 NiCrMoTiB 15 15 as well as the results of a previous intercomparison [2] are considered as validation that the thermophysical properties of the material investigated was determined within the given uncertainty intervals. Reliable relative expanded uncertainties have been stated for the properties determined and were achieved by applying good measurement practice, i.e., 3% for relative thermal expansion, 5% for specific heat capacity and thermal diffusivity, and between 6 and 8% for thermal conductivity. The mean values derived from this intercomparison agree well with the results of the previous one. Minor systematic deviations at high temperatures,

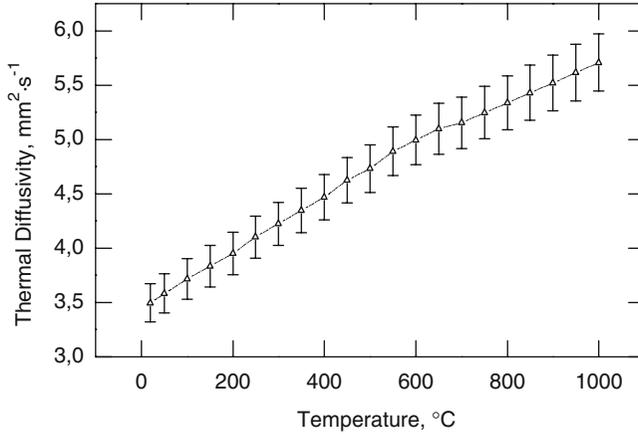


Fig. 5. Values derived for the non-weighted mean of the thermal diffusivity as a function of temperature.

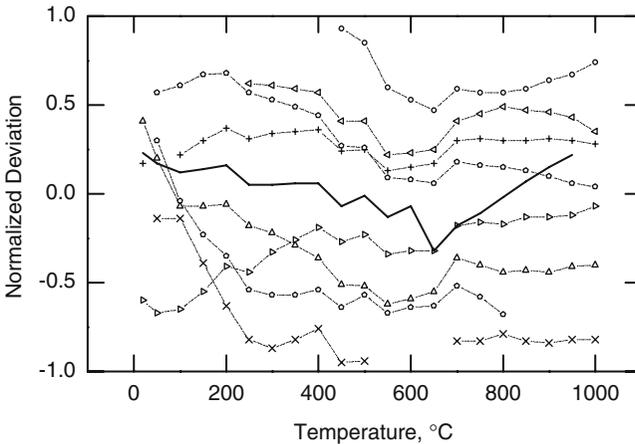
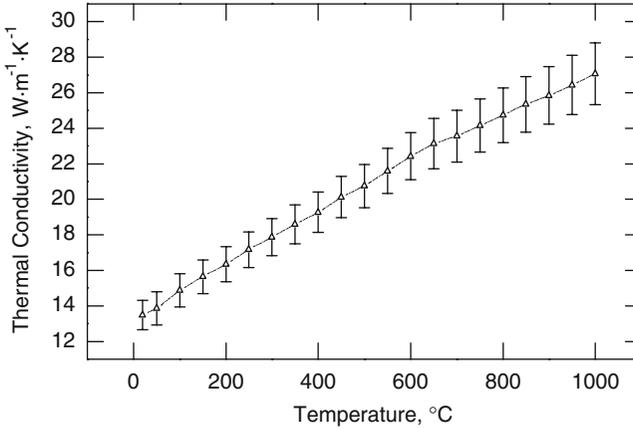
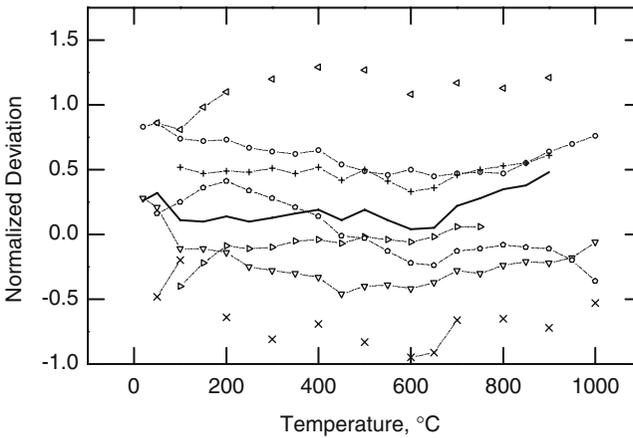


Fig. 6. Normalized deviation of the participating laboratories' thermal diffusivity results and of the previous intercomparison test [2] from the mean value derived as a function of temperature.

e.g., about 3% at 1000°C, were found in the case of the specific-heat-capacity measurements. This can be explained by the fact that the material is not in thermodynamic equilibrium. Therefore, pulse heating calorimetry and DSC give different heating-rate-dependent results for this material. A further reduction of the measurement uncertainties is limited by the stability of the material itself.



**Fig. 7.** Thermal conductivity calculated using the mean values of the specific heat capacity and the thermal diffusivity as a function of temperature.



**Fig. 8.** Normalized deviation of the participating laboratories' thermal conductivity results and of the previous intercomparison test [2] from the values calculated for the thermal conductivity as a function of temperature.

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