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Uncertainty of Thermal Diffusivity Measurements Using the Laser Flash Method[1]

L. Vozár[2, 3] and W. Hohenauer[4]

The paper deals with the uncertainty analysis for thermal diffusivity measurements using the laser flash method. A general metrological characterization of the high temperature thermal diffusivity measurement apparatus has been carried out. The metrological investigation follows the general rules for the evaluation and expression of uncertainty in measurement. This work presents a brief introduction to the flash method. It summarizes the main disturbing phenomena that may significantly influence the accuracy of the thermal diffusivity measurement. It gives a detailed description of the high temperature laser flash experimental apparatus installed at Austrian Research Centers. The paper also gives results of test measurements of the thermal diffusivity of a standard material, i.e., austenitic steel X10NiCrMoTiB1515 in the temperature range 20–900°C. The results are compared with literature data and discussed. Sources of measurement errors are analyzed; components of uncertainty are categorized according to their evaluation method. The results are subjected to rigorous statistical evaluation to determine the uncertainty associated with thermal diffusivity measurements.

KEY WORDS: austenitic steel; laser flash method; ISO GUM; thermal diffusivity; uncertainty analysis.

1. INTRODUCTION

A rather significant dispersion of results is a general feature of measurements of thermophysical properties for even well-defined solid materials.

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[2] Department of Physics, Faculty of Sciences, Constantine the Philosopher University, Tr. A. Hlinku 1, SK-94974 Nitra, Slovakia.
[3] To whom correspondence should be addressed. E-mail: vozar@nr.sanet.sk
[4] Department of Materials Research, ARC Seibersdorf Research GmbH, A-2444 Seibersdorf, Austria.

The current demand for accurate and reliable results has led to an understanding of the importance of the standardization of experimental methods and the development of standard materials providing reliable reference data for a wide range of experimental conditions.

The laser flash method [1] has become the most popular experimental method for measuring the thermal diffusivity. The simplicity and efficiency of the measurement, the accuracy and reliability of the results, and the possibilities of application under a wide range of experimental conditions and with a wide range of materials are the main advantages of the flash method. The fact that the flash method has meanwhile become the standard method in many countries confirms its universality. Its theory, operating principles, experimental aspects, and experimental data evaluation have been described in a large amount of scientific papers and reports (see review papers [2–6]).

The ARC Seibersdorf research GmbH uses a home-made laser flash experimental apparatus that has been continuously developed since its installation. To fulfill the current advanced requirements and fulfill the internationally accepted procedures and standards, attention has been focused on uncertainty analyses and metrological investigations.

This paper presents results of analyses of the reliability of thermal diffusivity measurements with the laser flash apparatus while providing an evaluated quantitative statement of the uncertainty. The analyses follow general rules for evaluating and expressing the uncertainty in measurement, established as the ISO GUM method (Guide to the Expression of Uncertainty in Measurement) — the method that has been adopted by various regional metrology and related organizations worldwide [7–9]. The GUM approach has been followed in expressing the estimated uncertainty of several thermophysical properties including thermal conductivity using the transient hot-strip technique [10], the guarded hot-plate technique [11], or the transient hot-wire method [12, 13]. The measurement results obtained for thermal diffusivity using the laser flash method have been metrologically evaluated [14, 15], and the uncertainty was systematically analyzed and expressed for different laser flash systems following the GUM recommendations [16].

2. FLASH METHOD

In the flash method the front face of a small disc-shaped sample receives a pulse of radiant energy coming from either a laser or a flash lamp. The thermal diffusivity is computed from the resulting temperature response on the opposite (rear) face of the sample. The simple ideal analytical model of the flash method is based on the thermal behavior

of a homogeneous, opaque, thermally insulated, infinite slab uniformly subjected to a short heat pulse of radiant energy over its surface. Assumptions of the model are as follows:

(a) the sample is homogeneous and isotropic, and the thermophysical properties and the density are uniform and constant and do not vary with temperature under experimental conditions,

(b) the sample is thermally insulated — there are no heat losses from the slab surfaces,

(c) the heat pulse is uniformly distributed over the slab surface and is absorbed by a material layer which is very thin in comparison to the sample thickness,

(d) the heat pulse is instantaneous, and its duration is negligible compared to the thermal response of the slab.

Under these assumptions the one-dimensional heat flow takes place across the slab. The shape of the rear-face temperature rise curve contains the information about the thermal diffusivity of the material. The conventional way to calculate the thermal diffusivity from the experimental data is that proposed by Parker et al. [1]. The method is rather simple; specifying the half time $t_{0.5}$, i.e., the time in which the rear-face temperature rise reaches half its maximum value, the thermal diffusivity is calculated from the expression,

$$a = 0.1388 \frac{e^2}{t_{0.5}}, \tag{1}$$

where e is the sample thickness.

Several other original data reduction methods (the algorithm for computing the thermal diffusivity from experimental data) in the flash method have so far been dealt with in the literature. They differ either in the analytical mathematical models used, or in the way the measured experimental rear-face temperature vs. time data are represented by a theoretical curve. A survey of the existing data reduction methods can be found elsewhere [17].

3. UNCERTAINTY OF MEASUREMENT RESULTS

Every measurement is affected by measurement errors that cause the difference between the measured value of the estimated property and its true value. The true value associated with the measured property is an

idealized notion, which cannot be determined. It is only an approximation or an estimate of the value measured [7–9].

The uncertainty of the result of a measurement generally consists of several components, which according to the GUM method may be grouped in two categories according to the method used to estimate their numerical values.

Type A standard uncertainties are evaluated by the statistical analysis of a series of observations. An evaluation may be based on any valid statistical method for treating data, i.e., calculating the standard deviation of the mean of a series of independent observations; using the method of least squares to fit a curve to data in order to estimate the parameters of the curve and their standard deviations; and then carrying out an analysis of the variance in order to identify and quantify random effects in certain kinds of measurements.

A Type B evaluation of the standard uncertainty is usually based on scientific judgment using all relevant information available, which may include previous measurement data; experience with, or general knowledge of, the behavior and properties of relevant materials and instruments; manufacturers specifications; data provided in calibrations and other reports; and uncertainties assigned to reference data taken from handbooks.

Each uncertainty component is represented by an estimated standard deviation, the standard uncertainty u_i, and is equal to the positive square root of the estimated variance u_i^2.

An uncertainty component obtained by a Type A evaluation is represented by a statistically estimated standard deviation, equal to the positive square root of the statistically estimated variance and the associated number of degrees of freedom.

In a similar manner, an uncertainty component obtained by a Type B evaluation is represented by a quantity u_j, which may be considered an approximation to the corresponding standard deviation; it is equal to the positive square root of u_j^2. It may be considered as an approximation to the corresponding variance and obtained from an assumed probability distribution based on all information available. Since the quantity u_j^2 is treated like a variance and u_j like a standard deviation, the standard uncertainty for such a component is simply u_j.

All the individual uncertainties influence the uncertainty of the resultant measurements. The combined standard uncertainty u_c represents the estimated standard deviation of the result. It is obtained by combining the individual standard uncertainties u_i arising from a Type A or a Type B evaluation using the usual method for combining standard deviations

based on the law of uncertainty propagation. Multiplying the combined uncertainty by a coverage factor k (typically in the range from 2 to 3), one obtains the expanded uncertainty U. It is confidently believed that the measurand Y (the true value of the measured property) is greater than or equal to $y-U$, and is less than or equal to $y+U$ (i.e., $Y=y\pm U$), where y is the measured value of the estimated property, i.e., the thermal conductivity. When the normal distribution applies, $U=2u_c$ (i.e., $k=2$) defines an interval having a level of confidence of approximately 95%, and $U=3u_c$ (i.e., $k=3$), it defines an interval with a level of confidence greater than 99%.

4. EXPERIMENTAL APPARATUS

The laser flash apparatus is regularly used for measurements of the thermal diffusivity of solids in the Materials Research Division of the Austrian Research Centre in Seibersdorf. It consists of an Nd:Cr:GGG (neodymium-doped gallium–gadolinium garnet) glass laser (BLS400, Baasel Lasertech) working at a wavelength $\lambda = 1.064\,\mu m$. The pulse energy is usually set to 5 to $6\,J\cdot cm^{-2}$ to keep the sample temperature rise below 3°C. The transient temperature is measured with a liquid nitrogen cooled HgCdTe infrared detector (HCT-80, Infrared Associated, Inc.) using a preamplifier (PPA-15-DC). The detector has a time constant of about 300 ns and is set to detect radiation from the central square area ($\sim 4\,mm^2$) on the sample rear face. The sample is held in a horizontal position in the vacuum chamber. A short tantalum tube acts as the resistance heater and allows measurements in the temperature range from 20 up to 1900°C. The furnace is powered by dc current from the power source (TN 10-5000, Heinzinger Elektronik). The sample temperature sensor consists of a steel encapsulated K-type (NiCr/Ni) thermocouple 1 mm in diameter, or a spot-welded S-type (Pt/PtRh10) thermocouple made from wires 0.35 mm in diameter (Heraeus). The data acquisition and control is performed using standard measurement hardware and a personal computer (pc).

The apparatus is constructed along two axes (Fig. 1), the laser being placed horizontally. The laser beam is reflected off a bending mirror and passes vertically through a glass window (BK7) into a water cooled stainless steel vacuum chamber. The vacuum is stabilized using a turbo pump (TPH 110, Pfeiffer Wakuumtechnik) at values of the order of 10^{-5} Pa. The sample holder consists of three molybdenum rods fixing the sample in a horizontal position in the central zone of the furnace. The construction allows irradiation of the lower (front) face of the sample and the measurement of temperature and temperature response on the upper (rear) face of the sample. The detachable top of the vacuum chamber fixes the IR

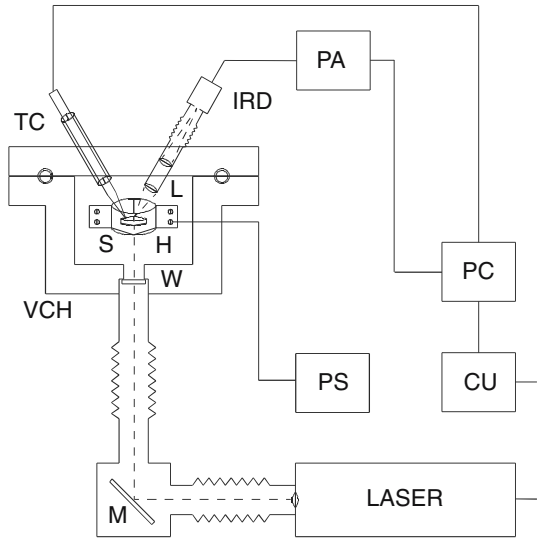

Fig. 1. Schematic view of the experimental apparatus: TC: thermocouple; IRD: infrared detector; PA: preamplifier; L: lens; S: sample; H: heater; W: window; VCH: vacuum chamber; M: mirror; PS: power source; PC: personal computer; CU: controller unit.

temperature sensor that is focused with a CaF_2 lens and mechanical iris. The chamber top contains the movable tubes that allow the thermocouple position to be set and, through a window, to be checked.

5. THERMAL DIFFUSIVITY OF STEEL

To determine the performance characteristics and the reliability of the equipment, various test measurements were performed on a stable and well-characterized specimen. The austenitic steel X10NiCrMoTiB1515 (Nr.1.4970) — a material that had been intensively investigated by the German Thermopysical Society [18] — was chosen for the experimental investigation. The composition of the material fully conforms to DIN (Table I). All measurements were performed in a vacuum.

As the material's thermal expansion during the measurement introduces a source of significant error, the sample thicknesses measured at room temperature were corrected according to results of performed dilatometric measurements of the test material. The thermal expansion values were measured in the temperature range from 20–900°C using a

Table I. Chemical Composition of the Austenitic Steel X10NiCrMo-TiB1515 (Nr.1.4970)

Element	Composition (mass%)
C	0.09
Si	0.45
Mn	1.7
P	0.003
S	0.004
Cr	14.6
Ni	15.0
Mo	1.25
Ti	0.46
Cu	0.07
B	0.0015
Al	<0.006

push-rod dilatometer (Netzsch 402C). The uncertainty analysis shows that the typical uncertainty of $\Delta L/L$ measurements is better than 1.5% of a measured value.

Table II presents results of three different measurements of the thermal diffusivity. Here the experimental readings are analyzed using Eq. (1) and applying the Clark and Taylor correction [19] for heat loss elimination. The values of a_1 to a_3 represent averages obtained from three different measurements. Table III summarizes the thermal diffusivity estimated values calculated using the data reduction procedure proposed by Degiovanni [20]. The method takes into account heat losses and gives three thermal diffusivity values computed for four different fractional times. The adiabatic limit temperature, i.e., the temperature the sample reaches after application of the laser heat pulse, is taken as the temperature of the measurement in order to eliminate the influence of variation of thermophysical properties with temperature [21] We see that the deviations in both cases lies between $+/-1.3\%$, which means that the reproducibility of the measurement in the temperature range between room temperature and 900°C is better than 1.3%, and the results practically do not depend on the data reduction procedure used. Table IV and Fig. 2 summarize these results for estimation of the thermal diffusivity of austenitic steel. Here a_{mean} is the mean value of the thermal diffusivities a_{01} and a_{02}, i.e., the average values taken from Tables II and III, and a_{REF} is the reference value taken from round-robin test measurements consisting of 10 independent measurements at six laboratories whose results were published in Ref. 18. These results

Table II. Thermal Diffusivity of Steel Calculated Using Eq. (1) Applying the Clark and Taylor Correction [19] (Standard deviation of a measurement is better than 1.3%)

	Thermal diffusivity (10^{-6} m^2·s^{-1})			Mean (10^{-6} m^2·s^{-1})	Standard deviation	
T (°C)	a_1	a_2	a_3	a_0	(10^{-6} m^2· s^{-1})	(%)
30	3.53	3.57	3.52	3.54	0.024	0.69
100	3.74	3.79	3.69	3.74	0.048	1.28
200	3.98	3.91	3.96	3.95	0.033	0.84
300	4.18	4.18	4.23	4.20	0.029	0.69
400	4.41	4.45	4.45	4.44	0.019	0.44
500	4.66	4.69	4.67	4.67	0.020	0.42
600	4.90	4.95	4.96	4.94	0.034	0.68
700	5.03	5.02	5.07	5.04	0.026	0.52
800	5.34	5.33	5.34	5.33	0.006	0.10
900	5.47	5.51	5.51	5.49	0.025	0.46

Table III. Thermal Diffusivity of Steel Calculated Using Data Reduction Method of Degiovanni [20] (Standard deviation of a measurement is similar as in Table II, better than 1.3%)

	Thermal diffusivity (10^{-6} m^2·s^{-1})			Mean (10^{-6} m^2·s^{-1})	Standard deviation	
T (°C)	a_1	a_2	a_3	a_0	(10^{-6} m^2· s^{-1})	(%)
30	3.54	3.56	3.62	3.57	0.038	1.05
100	3.77	3.80	3.77	3.78	0.017	0.46
200	3.93	3.94	3.96	3.94	0.016	0.39
300	4.27	4.22	4.24	4.24	0.023	0.53
400	4.42	4.44	4.47	4.44	0.026	0.59
500	4.68	4.65	4.68	4.67	0.016	0.35
600	4.88	4.99	4.95	4.94	0.054	1.10
700	5.02	5.02	5.13	5.05	0.061	1.21
800	5.24	5.27	5.36	5.29	0.061	1.16
900	5.45	5.48	5.46	5.46	0.017	0.30

confirm an uncertainty of measurements better than 1.7% in the temperature range between room temperature and 900°C.

Table V presents results of 12 independent measurements performed on different samples. Here the thermal diffusivity a is the average value reached in these tests at each temperature. Each individual thermal diffusivity value is calculated as the average of three values derived using the Eq. (1) and applying the Clark and Taylor correction [19] applied for different partial time ratios. A comparison shows that differences between

Table IV. Thermal Diffusivity of Steel: Comparison of Measured Values Taken from Tables II and III and Reference Data [18] (See Fig. 2) (These results confirm an uncertainty of measurements better than 1.7% in the temperature range between room temperature and 900 °C)

	Thermal diffusivity (10^{-6} m^2·s^{-1})				Standard deviation	
T(°C)	a_{01}	a_{02}	a_{mean}	a_{REF}	(10^{-6} m^2·s^{-1})	(%)
30	3.54	3.57	3.56	3.57	−0.013	−0.37
100	3.74	3.78	3.76	3.75	0.009	0.24
200	3.95	3.94	3.95	3.99	−0.045	−1.13
300	4.20	4.24	4.22	4.24	−0.021	−0.50
400	4.44	4.44	4.44	4.48	−0.042	−0.95
500	4.67	4.67	4.67	4.73	−0.058	−1.24
600	4.94	4.94	4.94	4.98	−0.042	−0.85
700	5.04	5.05	5.05	5.10	−0.054	−1.06
800	5.33	5.29	5.31	5.33	−0.018	−0.34
900	5.49	5.46	5.48	5.57	−0.092	−1.67

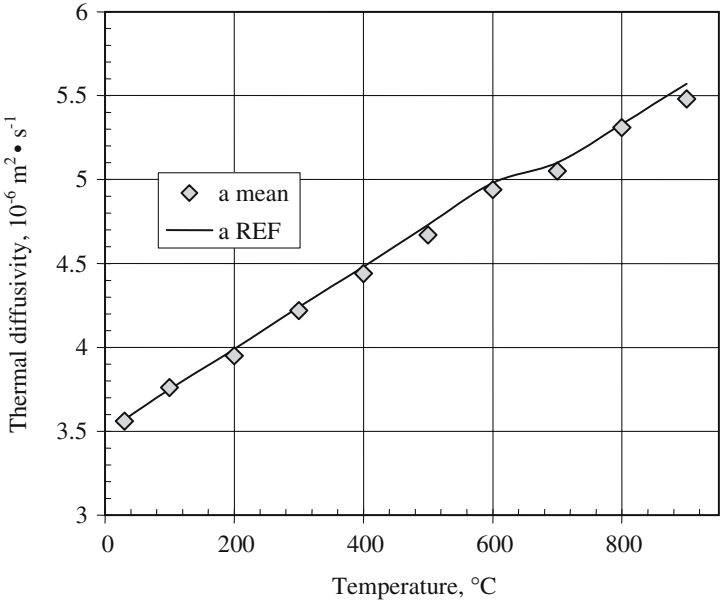

Fig. 2. Thermal diffusivity of steel. Comparison of measured values (a_{mean}) and reference data (a_{REF}) taken from Table IV.

Table V. Thermal Diffusivity of Steel: Comparison of Measured Values – a is the Average Obtained from 12 Independent Measurements on Different Samples and Reference Data [18]

	Thermal diffusivity					
T(°C)	a (10^{-6} m^2·s^{-1})	stdev (10^{-6} m^2·s^{-1})	stdev/a (%)	a_{REF} (10^{-6} m^2·s^{-1})	$a_{REF} - a$ (10^{-6} m^2·s^{-1})	$(a_{REF} - a)/a_{REF}$ (%)
30	3.56	0.037	1.03	3.57	−0.011	−0.30
100	3.75	0.038	1.02	3.75	−0.002	−0.04
200	3.99	0.042	1.04	3.99	−0.001	−0.03
300	4.23	0.038	0.89	4.24	−0.013	−0.30
400	4.48	0.034	0.76	4.48	−0.004	−0.09
500	4.72	0.026	0.55	4.73	−0.013	−0.27
600	4.99	0.023	0.47	4.98	0.005	0.10
700	5.11	0.031	0.61	5.1	0.012	0.24
800	5.34	0.049	0.92	5.33	0.014	0.26
900	5.55	0.031	0.56	5.57	−0.018	−0.33

the thermal diffusivities obtained and the reference values are less than 0.3%. The standard deviation is smaller than 1.03%. It generally decreases with an increase in temperature. This results from an increase in the sensitivity of the IR temperature detector with temperature.

6. ESTIMATION OF UNCERTAINTY

The estimation of the thermal diffusivity using the flash method is based on a knowledge of the sample thickness and of the shape of the temperature rise versus time evolution of the sample rear face. The sources of the measurement uncertainties are therefore linked with the sample itself, the temperature measurements, the performance of the detector and the data acquisition board as well as data analysis. Other phenomena — the finite pulse time effect, nonuniform heating, and heat losses from the sample — are also uncertainty sources.

6.1. Type A Component of Uncertainty

The relative standard deviation values given in Table V represent the Type A uncertainties involved in this measurement process. It can be concluded that the value of 1.1% represents the Type A component of the uncertainty associated with the thermal diffusivity measurements.

6.2. Type B Uncertainty Components

Type B uncertainty components are estimated and discussed individually for each uncertainty source. Values of these components are given as limits between which the particular influence quantity may generate a variation of the measured value. This means that a rectangular distribution is implicitly assumed for the occurrence probability of the values within the limits given.

6.3. Sample Thickness

The sample thickness is measured with a certified micrometer with an uncertainty of 0.5 μm. For the sample that typically is 2.5 mm thick, the relative uncertainty is 0.02%. The thermal diffusivity is related to the square of the specimen thickness and

$$\frac{\Delta a}{a} = 2\frac{\Delta e}{e}. \tag{2}$$

The error limits associated with the sample thickness measurement are 0.04%. This uncertainty is usually reduced when performing repeated measurements of the sample thickness and taking the average value into account.

The material's thermal expansion during the test introduces another error source that should be taken into account. Table VI presents results of dilatometric measurements of the test material. Thermal expansion values for the temperature range of 20–900°C show that the maximum relative prolongation is $\Delta L/L = 16.99 \times 10^{-3}$. The error caused when the results are not corrected for the thermal expansion of steel is therefore estimated to be smaller than 3.4%. Since the thermal expansion correction has been applied, we do not consider the error limits associated with the thermal expansion of the sample.

6.4. Absolute Sample Temperature

The absolute (steady-state) sample temperature does not enter into the thermal diffusivity determination. It is the temperature of the measured thermal diffusivity that is referenced. The temperature is measured using a Type K thermocouple (NiCr/Ni) whose uncertainty as specified by its manufacturer is 1.1°C or 0.4% of the measured value. The uncertainty of the thermal diffusivity depends on the thermal diffusivity versus temperature dependence of the measured material. Considering the steel thermal diffusivity versus temperature dependence, it can be stated that the

Table VI. Thermal Expansion of Austenitic Steel X10NiCrMoTiB1515 (Nr.1.4970) Measured Using Push-Rod Dilatometer (Netzsch 402C) with Uncertainty Better Than 1.5% of a Measured Value

T (°C)	$\Delta L/L\,10^{-3}$
50	0.52
75	0.95
100	1.39
125	1.84
150	2.29
175	2.75
200	3.21
225	3.68
250	4.14
270	4.52
300	5.09
350	6.05
400	7.03
450	8.02
500	9.02
550	10.02
600	11.04
650	12.07
700	13.11
750	14.13
800	15.13
850	16.08
900	16.99

uncertainty of the thermal diffusivity is less than 0.4% in the temperature range of 20–900°C.

It should be noted that during a thermal diffusivity experiment, the effective temperature of the sample is higher than its steady-state temperature before the (laser) heat pulse is applied. For the case where the thermal diffusivity of the measured material depends strongly on the temperature, which is not the case for steel, a special analysis should be performed [6].

The reliability of the thermal diffusivity measurement depends strongly on the stability with which thermal equilibrium is achieved within the sample before a measurement is started (before application of the heat pulse). A constant absolute temperature at $+/-1$°C over an interval of 3–5 min was found to be an acceptable condition. Our experience shows that it is very important to check the time stability of the relative temperature of the rear face. The relative temperature rise is measured

with much higher temperature resolution than the absolute temperature of the sample. We found that the changes of this temperature evolution — systematic or random — dramatically influence the reliability of the measurement. That is why the sample's rear face temperature is continuously monitored as in the measurement — with the same sensitivity of the temperature measurement and with the same sampling (working) frequency — and the time interval and the measurement (heat pulse application) starts only when the required temperature stability at the desired measurement temperature is achieved.

6.5. Rear Face Temperature Evolution — Temperature Detector Inertia

The manufacturer of the IR temperature detector specifies a time constant of 300 ns. As the typical response time is of the order of one tenth of a second (the half time for a 3-mm thick sample is about $t_{0.5} = 0.1$ s from 20 to 800°C), the uncertainty in the temperature measurement due to inertia (response time) of the temperature detector is extremely small. The influence of this phenomenon on the accuracy of the thermal diffusivity measurement can therefore be neglected.

6.6. Nonlinearity of the Temperature Detector

In the measurement it is important to ensure that the temperature detector operates within the linear range where the signal response of the detector (voltage) is directly proportional to the input radiation. This is valid for small temperature changes (smaller than 10°C) [17]. According to Planck's law, within this small temperature change nonlinearities are less than 1% for temperatures up to 1000°C. In our measurements the rear face temperature increase is always kept below 3°C by setting a suitable laser power that generates the appropriate heat pulse over the front face of the sample.

The nonlinearity of the preamplifiers and the analog-to-digital conversion represents the second source of possible nonlinearities of temperature measurement. The manufacturer of the data acquisition board ensures that the linearity of the preamplifier at the gain used in the measurement as well as the linearity of the analog-to-digital conversion are better than 0.065%.

As the thermal diffusivity determination is based on an analysis of the temperature rise versus time curve, the influence of the nonlinearity of the temperature rise detection can be reduced or minimized by utilizing the "whole" temperature versus time curve rather than only a single

point in the data reduction. Moreover, the reliability of our measurements is checked by comparing the measured temperature rise versus time curve with the analytical curve as well as by analyzing the experimental data with several data reduction procedures. In this way, any potential deviations of the experimental conditions from those assumed in the analytical model can be easily identified. We estimate that the influence of the nonlinearity of the temperature rise measurement on the uncertainty of the thermal diffusivity measurement is less than 0.5%.

6.7. Performance of the Digital Data Acquisition Board

The manufacturer has stated the following characteristics of the data acquisition board used here:

Signal resolution: 12 bit (1 in 4,096, or 0.02%)

A/D conversion time: 706 μs (at maximal gain—1000)

Time base uncertainty: 0.015%

Based on these characteristics, it is considered that the errors of thermal diffusivity due to the operating errors of the digital data acquisition board are negligibly small compared to the other error sources.

6.8. Time Scale and the Time Origin

Since the measurement of thermal diffusivity is based on an analysis of temperature rise versus time evolution, the accuracy and reliability of the time scale is essential for the accuracy of the thermal diffusivity measurement. In our measurement the time scale is derived from an 8-MHz quartz-based timer whose time base stability is stated by the manufacturer to be better than 0.015%.

The time origin is measured using a fast photodetector (photodiode) that measures the laser light inside the laser cell. The photodetector has a response time of the order of μs. The time scale is set to perform 1000 measurements in a desired time interval. For the steel measurement, the working frequency was set to 2 kHz. We estimate that the error associated with the time origin measurement is therefore 0.5 ms, i.e., the time scale may be shifted by that amount. As the half time for a 3-mm thick steel sample is about $t_{0.5} = 0.1$ s from 20 to 900°C when evaluating the measurement using Parker's procedure (Eq. (1)), the uncertainty in the thermal diffusivity estimation should be less than 0.5%. When the thermal diffusivity is estimated using more sophisticated data reduction methods, the influence of this phenomenon should be much smaller.

6.9. Heat Pulse Width

The photodetector measures the onset of the laser flash. To ensure consistency with theory, the heat pulse shape and width should be taken into account. The manufacturer states that the laser flash duration is 0.2–1.5 ms. To take the heat pulse width into account, the time origin is shifted to the center of gravity using the procedure described in Ref. 22. If one compares the correction time with the half-time value, it can be seen, that correcting the data for the pulse width can have an influence of 0.75%. We estimate that the influence of the heat pulse width on the uncertainty of the thermal diffusivity measurement is smaller than 0.1%.

6.10. Nonuniform Pulse Heating

The uniformity of the laser beam is directly related to the uniformity of sample heating, and is usually a major source of error. The manufacturer specifies homogeneity of the laser beam within a diameter of 12 mm. We use only the central part of a diameter of 10 mm. To ensure and check the homogeneity of the laser pulse, a sheet of photographic paper is periodically exposed to the laser light. The homogeneity of the absorption surface is ensured as well. Here we do not evaluate the influence of the non-uniformity of pulse heating on the uncertainty of the thermal diffusivity measurement.

6.11. Heat Losses

To eliminate heat losses to the greatest possible extent, several improvements of the sample holder and experimental cell were made. Heat loss between the sample and the environment cannot be neglected. That is why we utilize the data reduction methods accounting for heat losses. Agreement among the thermal-diffusivity values obtained using different data reduction methods assures the reliability of the measurements. Taking into account the results from Tables II and III, we estimate that the influence of the heat losses on the uncertainty of the thermal diffusivity measurement is smaller than 1.3%.

Table VII summarizes the estimates of the uncertainties according to their type and source. Since we assume a uniform (rectangular) distribution, the corresponding individual standard deviations can be calculated from the equation,

$$u_j = a/\sqrt{3}, \tag{3}$$

Table VII. Sources of Uncertainties and Standard Deviations

Type of uncertainty	Source of uncertainty	Uncertainty limits (%)	Standard deviation (%)	
A	Random		1.1	1.1%
B	Sample thickness	0.04	0.023	0.89
	Sample thermal expansion	0	0	
	Absolute sample temperature	0.4	0.231	
	Temperature detector inertia	0	0	
	Nonlinearity of temperature detector	0.5	0.289	
	Performance of digital data acquisition board	0	0	
	Time scale and time origin	0.5	0.289	
	Heat pulse width	0.1	0.058	
	Nonuniform pulse heating	x		
	Heat losses	1.3	0.751	

where $a = (a_+ - a_-)/2$ and a_+ and a_- are the estimated upper and lower limits.

Since we assume that there is no correlation among the uncertainty sources, the Type B component of the thermal diffusivity measurement uncertainty can be calculated as the square root of the sum of the squares of the individual standard deviation values. This leads to a 0.89% Type B uncertainty being associated with the experiments. The combined standard uncertainty is calculated similarly from the Type A and B components. With a 1.1% Type A uncertainty, the combined standard uncertainty becomes 1.99%. The expanded uncertainty (with $k = 2$) gives then a value of 3.98% within a 95% confidence level.

7. CONCLUSIONS

This paper presents results of an uncertainty analysis of thermal diffusivity measurements using the laser flash method apparatus installed at the ARC Seibersdorf research GmbH through a series of test measurements performed on austenitic steel X10NiCrMoTiB1515 (Nr.1.4970). It is concluded that the expanded uncertainty associated with the thermal diffusivity measurement in the temperature range from 20 to 900°C is 3.98% within a confidence level of 95%.

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