International Comparison on Thermal-Diffusivity Measurements for Iron and Isotropic Graphite Using the Laser Flash Method in CCT-WG9

M. Akoshima $\,\cdot\,$ B. Hay $\,\cdot\,$ J. Zhang $\,\cdot\,$ L. Chapman $\,\cdot\,$ T. Baba

Received: 14 October 2011 / Accepted: 4 May 2012 / Published online: 3 June 2012 © Springer Science+Business Media, LLC 2012

Abstract The first international pilot study of thermal-diffusivity measurements using the laser flash (LF) method was organized by the working group 9 (WG9) of the Consultative Committee for Thermometry (CCT) of the Bureau International des Poids et Mesures (BIPM). Four National Metrology Institutes (NMIs) participated in this comparison. Thermal-diffusivity measurements on the Armco iron and the isotropic graphite IG-110 were carried out from room temperature to about 1200 K. The sample sets consist of five disk-shaped specimens of 10 mm in diameter and (1.0, 1.4, 2.0, 2.8, and 4.0) mm in thickness, each cut from the same block of material. These sample sets were specifically prepared for the comparison and sent to the participants. In the pilot comparison, the thermal diffusivity of each sample was estimated using the LF method with a specific extrapolating procedure. This procedure has the advantage of determining the inherent thermal diffusivity values versus the amplitude of the output signal corresponding to the temperature rise during each measurement is defined as

M. Akoshima (⊠) · T. Baba National Metrology Institute of Japan (NMIJ), AIST, Japan, Tsukuba Central 3, 1-1-1 Umezono, Tsukuba, Ibaraki, 305-8563, Japan e-mail: m-akoshima@aist.go.jp

B. Hay

J. Zhang National Institute of Metrology (NIM), Bei San Huan Dong Lu 18, Beijing 100013, China

L. Chapman National Physical Laboratory (NPL), Teddington, Middlesex, TW11 0LW, UK

Scientific and Industrial Metrology Centre, Laboratoire National de Métrologie et d'Essais (LNE), 1 rue Gaston Boissier, 75015, Paris, France

the inherent thermal diffusivity. The overall results showed good agreement between independent laboratories, measurement equipment, and specimen thicknesses. The thermal diffusivities of the materials were determined using our measured results. A quantitative evaluation of the variability of the data obtained by the participants has been done, by evaluating the deviations from the reference value, the *Z*-value, and the *En*-number. Some data showed a large deviation from the reference value. It was concluded that these are caused by an insufficient time response of the measurement equipment and some difficulties with changing the pulsed heating energy. The effect of the thermal expansion on the thermal diffusivity was checked. It was found that the thermal-expansion effect was very small and negligible in this case.

Keywords Thermal diffusivity · Laser flash method · International comparison · Graphite · Iron · National metrology institutes

1 Introduction

Thermophysical properties of solids are interesting because of enhancing the performance of various apparatus, electronic devices, and home electronics. There is a trend for the expectation of reliability and traceability in the field of thermophysical property measurements. In general, the reliability in the field of measurement is estimated quantitatively by uncertainty evaluation. Traceability in the field of measurements means to be traceable to the International System of Units (SI units) [1] supplied by Bureau International des Poids et Mesures (BIPM) [2] managed by Comité International des Poids et Mesures (CIPM) [3]. The value which satisfies both of these terms has a meaning of the absolute value.

The laser flash (LF) method [4] is a well-known method to measure the thermal diffusivity of solids. The thermal diffusivity of solids of a typical size of about 1 mm to 5 mm in thickness may be measured using this method from room temperature to well above 1000 K. Thermal-diffusivity values measured by this method are widely used in industry. Various types of apparatus and many varieties of data analysis procedures are used to calculate thermal-diffusivity values from temperature-rise curves for the LF method. The method is very popular for practical measurements. There is a need to confirm the reliability of thermal diffusivities measured by the LF method since the thermal problem is a hot topic in view of energy-saving and enhancing performance in various applications. The LF thermal-diffusivity measurement can be evaluated analytically because the principle of the method is very simple, one-dimensional heat diffusion phenomena. Thus, the method is preferable to use as the standard method to obtain the thermal diffusivity. In fact, the National Metrology Institute of Japan (NMIJ) and the Laboratoire National de Métrologie et d'Essais (LNE) have established the SI traceable thermal-diffusivity measurement and the uncertainty evaluation as the national standard [5,6]. NMIJ is supplying the reference material using the technique [7].

Thermophysical properties are discussed in the Working Group 9 of the Consultative Committee for Thermometry (CCT-WG9) [3] of CIPM. The term of reference of CCT-WG9 is "to advise the CCT on matters related to thermophysical properties, and to assess the need in this subject field for a key comparison." The CCT-WG9 members discuss thermophysical properties with respect to the standard and application. Three kinds of international comparisons were carried out within this community: thermal-conductivity measurements of insulating materials using the guarded hot-plate method (CCT-P01), thermal-diffusivity measurements of solids using the LF method (CCT-P02), and normal spectral emissivity measurements of solids (CCT-P03).

The first LF thermal-diffusivity comparison was proposed at the CCT-WG9 Meeting in September 2005 in Bratislava, Slovakia. We discussed the plan for this comparison including the schedule, samples, and measurement conditions at the WG9 meeting in August 2006 in Boulder, CO, USA. LNE, the National Institute of Metrology (NIM), NMIJ, and the National Physical Laboratory (NPL) agreed to participate in the comparison. NMIJ was the pilot laboratory of this comparison. The samples for the comparison were sent to these participants in January 2007, and the measurements were started at each laboratory. The measurements were carried out over a period of about 10 months. The measured data were sent to the pilot in February 2008. The preliminarily results was summarized by the pilot and reported at the WG9 meeting in May 2008. We also showed the preliminary results at an international conference [8]. After the continuous discussions and the data evaluation, we summarized the final results of the comparison in 2011.

The objective of this comparison was to investigate the state of the art for thermaldiffusivity measurements using the LF method in NMIs and to find common understanding about measurement procedures, data analysis procedures, and the evaluation of uncertainty in thermal-diffusivity measurements.

Figure 1 shows the principle of the LF method. The surface of the specimen is uniformly heated by pulsed flash light from a lamp or laser. The heat at the surface diffuses throughout the whole specimen. This phenomenon is observed as the temperature-rise curve, the time dependence of the rear surface temperature observed by an infrared radiometer. The thermal diffusivity α is obtained from the sample thickness *d* and the heat diffusion time τ_0 calculated from the observed temperature-rise curve as follows [4]:



Fig. 1 Principle of the LF method

$$\alpha = \frac{d^2}{\tau_0} \tag{1}$$

This study, which is an inter-comparison of NMIs, aims to discuss thermal-diffusivity measurements from the viewpoint based on the SI units and inherent material properties. The thermal diffusivity is just one of the physical properties dependent on temperature. It is given as a function of length and time in the case of the LF method. Thermal diffusivity is a derived quantity consisting of length, time, and temperature. Therefore, thermal diffusivity can be traced back to the SI units [5]. In the field of metrology, uncertainty evaluations based on the "Guide to the expression of uncertainty in measurement" (GUM) [9] are preferable.

It is considered that the thermal diffusivity is a property inherent to the material. According to this, the thermal diffusivity does not depend on measurement conditions, shape, and size. However, it is known that measured results are often influenced by these factors. The procedure to obtain the inherent thermal diffusivity was proposed [10]. The temperature of the sample changes from the initial temperature to the finite temperature due to the pulsed heating of the surface during the measurement. Since the thermal diffusivity depends on temperature, the value calculated from a temperature-rise curve that includes the influence of thermophysical properties from the initial temperature to the finite temperature is the apparent thermal diffusivity. The inherent thermal diffusivity at the initial temperature is expected to be the value calculated from a temperature-rise curve with zero-temperature rise. Then measurements were carried out changing the pulsed heating energy at a stable temperature. The apparent thermal-diffusivity values calculated from each temperature-rise curve were plotted against the temperature rise consistent with the pulsed energy as shown in Fig. 2. The inherent thermal diffusivity at the temperature is determined as the extrapolated value to the zero-temperature rise in the plot.

The sample thickness is also one of the measurement conditions. According to Eq. 1, the heat diffusion time depends on the sample thickness because the thermal



Fig. 2 Principle of the extrapolating procedure (example of measurements of isotropic graphite sample at room temperature, $T_i = 298$ K)

diffusivity is inherent in the material. By measuring various thicknesses of samples cut from a block of solid, at the same temperature and changing the pulsed heating energy, we will confirm the agreement of the extrapolated value in the plot of the amplitude of the output signal dependence with apparent thermal-diffusivity values.

2 Experimental

2.1 Samples

Metals and ceramics are usually measured using this method. There is a need to discuss the effect of surface treatment. According to them, two materials were selected for this pilot study: Armco iron and IG-110, a grade of isotropic graphite. Both materials are dense, homogeneous, and chemically stable solids. The thermal diffusivity of these materials were measured at the beginning of the comparison by NIMJ (for IG-110 isotropic graphite) and LNE (for Armco iron) for all the sets of specimens in order to check their homogeneity. The Armco iron is a metal which shows a middle range value of the thermal diffusivity of solids. It needs some treatment, for example, sand-blasting and coating of the surfaces, to measure the thermal diffusivity by the LF method. The sample sets of the Armco iron were supplied by LNE for the comparison. The isotropic graphite is a popular carbon material and shows reasonably high values for the thermal diffusivity. The isotropic graphite is a convenient material for the LF method because it is not necessary to coat the sample surfaces. It is a good candidate material for this comparison because the thermal diffusivity shows a strong dependence with temperature. IG-110 is well-characterized by NMIJ as the certified reference material (CRM) [7]. The IG-110 sample sets made from the different lot of the CRM were supplied by NMIJ for the comparison.

The samples are disks of 10 mm in diameter. They consist of a set of disks with 1.0 mm, 1.4 mm, 2.0 mm, 2.8 mm, and 4.0 mm thicknesses, from an adjacent position from a block in order to confirm that the thermal diffusivity is independent of thickness. The set of specimens for a participant is as shown in Fig. 3.

2.2 Participants and Apparatus

Four NMIs have participated in this LF comparison. The measurement systems, conditions of use, and analysis methods of each participant are described in Table 1. Laboratory 1 and Laboratory 4 have their homemade measurement systems. Laboratory 2 has a system which was developed using some technical advantages [16].



Fig. 3 Sample sets of (a) Armco iron and (b) isotropic graphite for this comparison

Participant	Apparatus	Pulsed heat source	Rear surface observation	Atmosphere	Analysis method
Lab.1	Homemade [6]	Nd-glass (1054 nm)	InSb sensor MCT sensor	Ar flow	Partial-time moment method [11]
Lab.2	LFA-502N (customized model for the standard)	Nd-YAG (1064 nm)	InSb sensor	Vacuum	Curve fitting with equal- area method [13–15]
Lab.3	LFA-427 (commercial model)	Nd-YAG (1064 nm)	InSb sensor	Ar flow	Curve-fitting method [13,14]
Lab.4	Homemade	Nd-YAG (1064 nm)	Thermocouple	Ar flow	Half-time method [4]

 Table 1
 Participants and their apparatus, measurement conditions, and analysis methods

The apparatus of Laboratory 3 is a manufacturing model. Laboratory 1 and Laboratory 2 have SI traceable thermal-diffusivity measurement techniques.

2.3 Measurement Procedure

The measurements were carried out during two heating cycles from room temperature (RT) to 1200 K as follows: RT (300 K), 600 K, 900 K, 1200 K, RT (300 K), 600 K, 900 K, 1200 K, and RT (300 K), respectively.

For each temperature, the pulsed heating energy dependence of the apparent thermal diffusivity was investigated to determine the inherent thermal diffusivity. The apparent thermal diffusivity and the temperature rise were estimated for each temperature-rise curve. The apparent thermal-diffusivity values were plotted against the temperature rise consistent with the pulsed energy. The inherent thermal diffusivity at the temperature test (initial temperature) was determined as the extrapolating value to the zero-temperature rise in the plot as shown in Fig. 2.

2.4 Uncertainty Evaluation

The uncertainty of the thermal diffusivity was reported from each participant with the measured thermal diffusivity. The uncertainty evaluation was carried out according to the respective method [6, 10, 17]. Laboratory 3 had some difficulties with evaluating the uncertainty in this study.

3 Results and Discussion

The thermal diffusivity of Armco iron measured by the participants is shown in Fig. 4. The participants are identified by Lab.1, Lab.2, Lab.3, and Lab.4. The data of the samples with (1.0, 1.4, 2.0, 2.8, and 4.0) mm thicknesses are plotted as (1), (2), (3),

(4), and (5), respectively. So, the data labeled "Lab.1(1)" is the value measured by the participant "Lab.1" on the sample with 1.0 mm thickness. The thermal diffusivity reported from Lab.1, Lab.2, and Lab.3 in Fig. 4 was determined using the extrapolating procedure. The thermal diffusivity of Lab.4 is the average of the measured data with three levels of the pulsed heating energy since it was difficult to determine the value by the extrapolating procedure using the three levels of the data. It was difficult to determine the temperature dependence of the thermal diffusivity of Armco iron in the range from 300 K to 1200 K from these values obtained at four temperature levels, because this material presents a magnetic phase transition at about 1043 K. LNE proposed therefore to use the following temperature dependence based on more detailed measurements [18]:

$$\alpha (T) = 2.12 \times 10^{-5} - 4.06 \times 10^{-8}T + 2.45 \times 10^{-11}T^2 - 2.06 \times 10^{-17}T^4$$

$$(293 \text{ K} < T < 1043 \text{ K})$$

$$\alpha (T) = -1.172 \times 10^{-2} + 5.41 \times 10^{-5}T - 9.36 \times 10^{-8}T^2 + 7.20 \times 10^{-11}T^3$$

$$-2.07 \times 10^{-14}T^4$$

$$(T > 1043 \text{ K})$$

$$(3)$$

The results from participants agree with these functions as shown in Fig. 4.

Figure 5 shows the determined thermal diffusivity for IG-110. The thermal-diffusivity values obtained by the participants agreed with each other independent of the participant and specimen thickness. The temperature dependence proposed by NMIJ [10] was determined by fitting to these values as follows:



Fig. 4 Thermal diffusivity of Armco iron reported by the participants



Fig. 5 Thermal diffusivity of isotropic graphite reported by the participants

$$\alpha(T) = -3.30 \times 10^{-5} + 3.66 \times 10^{-5} \exp\left(\frac{382}{T}\right)$$
(4)

Figure 6 shows deviations from the temperature dependence curve plotted with uncertainty (coverage factor k = 1). The uncertainty from Lab.3 is not yet reported. The uncertainty (k = 2) is typically reported as about 2 % to 4 % from Lab.1, Lab.2, and Lab.3 as error bars show in Fig. 6. The uncertainties were estimated by each laboratory. The typical common uncertainty factors were uncertainty due to sample thickness, uncertainty due to time scale of temperature-rise curve observation, uncertainty due to pulse heating duration, uncertainty due to non-uniformity of the laser beam, uncertainty due to temperature stability during measurements, and uncertainty due to analysis. In both cases of Armco iron and IG-110, the deviation was very small at room temperature.

These deviations are smaller for the Armco iron than for IG-110. It was found that it might be difficult to measure high thermal-diffusivity materials due to the time response of the measurement system. Since the thermal diffusivity of IG-110 is larger than that of Armco iron, measurement systems are needed to have a fast time response to measure IG-110 compared with the case of Armco iron. The thin thickness samples (1.0 mm and 1.4 mm) were difficult to measure for the same reason. According to Figs. 4, 5, and 6, it seems that the range of the thermal diffusivity between $0.5 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ and $2.0 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ is easy to measure for all participants. This result is very important for the next comparison.

Figures 7 and 8 show an example of measured apparent thermal-diffusivity values by changing the pulsed heating energy and the inherent thermal diffusivity estimated at room temperature by each participant using the extrapolating procedure. In the case of the Armco iron specimen with 2.8 mm thickness, as shown in Fig. 7, apparent



Fig. 6 Deviations from the temperature dependence curve on Armco iron (a–d) and IG-110 (e–h) at each temperature. Symbols show data for 1.0 mm ($\mathbf{\nabla}$), 1.4 mm ($\mathbf{\bullet}$), 2.0 mm (\boxplus), 2.8 mm ($\mathbf{\Delta}$), and 4.0 mm (\Box) thick samples. Error bars show uncertainties of the data

thermal-diffusivity values range from $1.97 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ to $2.03 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ for Lab.1, from $2.00 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ to $2.18 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ for Lab.2, from $1.91 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ to $2.37 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ for Lab.3, and from $2.05 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ to $2.07 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ for Lab.4. It is found that the amplitude of the output signal varies

Deringer



Fig. 7 An example of measured apparent thermal-diffusivity values changing the pulsed heating energy and the inherent thermal diffusivity of the Armco iron estimated by the extrapolating procedure at room temperature



Fig. 8 An example of measured apparent thermal-diffusivity values changing the pulsed heating energy and the inherent thermal diffusivity of IG-110 estimated by the extrapolating procedure at room temperature

between the participants because the amplitude value depends on the measurement system and the measurement conditions. These apparent thermal-diffusivity values scatter mainly between $1.9 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ to $2.3 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$. The deviation is about 20%. The inherent thermal diffusivity determined using the extrapolating procedure is determined within 8% deviation. In the case of the IG-110 specimen with 2.8 mm thickness, the apparent thermal-diffusivity values scatter between $8.7 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$ to $10.1 \times 10^{-5} \text{ m}^2 \cdot \text{s}^{-1}$, as shown in Fig. 8. The deviation is about 14%. The deviation of the inherent thermal diffusivity estimated by the extrapolating procedure is almost the same or a little bit smaller than this. Thus, the extrapolating procedure is effective to determine the inherent thermal diffusivity with a small deviation independent of the measurement systems and measurement conditions.

The thermal diffusivity is calculated from the specimen thickness and heat diffusion time as shown in Eq. 1. The measurements were carried out from 300 K to 1200 K. The heat diffusion time was estimated from each temperature-rise curve measured at each temperature. The specimen thickness was measured around 300 K by a micrometer. In general, the thickness may change because the thermal expansion of a solid material depends on temperature. We have to make a correction if the thermal expansion of the specimen is larger than the uncertainty of the thermal-diffusivity measurement.

Thermal-expansion measurements were carried out by Lab.1 using a push-rod dilatometer. The results are as shown in Table 2. The thermal-expansion correction for the thermal diffusivity calculated from the thermal-expansion coefficient is about 0.8% in the case of Armco iron. That of IG-110 is about 0.3%. It can be almost ignored

Temperature (K)	Thermal-expansion coefficient (10^{-6} K^{-1})		Thermal-expansion correction for thermal diffusivity	
	Armco iron	IG-110	Armco iron	IG-110
296	_	_	_	_
600	12.9	3.8	1.0079	1.0023
900	14.1	4.3	1.0086	1.0026
1200	12.2	4.7	1.0074	1.0029

 Table 2
 Thermal-expansion coefficient of Armco iron and isotropic graphite measured by push-rod dilatometer

because uncertainties of thermal-diffusivity measurements were about 3 %. The thermal-expansion effect is not large in this study.

In order to study the deviation of these measured thermal-diffusivity values in detail, we calculated the *Z*-value and *En*-number. We can check the performance of the measured results according to ISO/IEC 17043 Annex A [19].

The Z-test is a statistical method used to test the normal distribution. This method aims to test whether the differences in results are statistically significant for the sample mean and population mean. (It enables us to examine whether the difference between the average of results for a sample and the average of results for the whole population is significant or not, in the statistics authorization method that uses a normal distribution.) The Z-value is expressed as follows:

$$Z = \frac{x - X}{s} \tag{5}$$

Here, *x* is the measured value and *X* is the mean value, for example, the average of measured values and values of fitted function *s* is the estimated variance, for example, the standard deviation of *X*. $|Z| \leq 2$ indicates "satisfactory" performance. In the case of " $2 < |Z| \leq 3$," the data are questionable. And outlier results show |Z| > 3. Figure 9 shows *Z*-values of our measured thermal diffusivity.

The *En*-number is derived by dividing the difference between a participant's test data and the test artifact's assigned value by the square root of the sum of the squares (RSS) of the participant laboratories' test data uncertainty and the reference laboratory's test artifact's assigned value uncertainty.

$$En = \frac{x - X_{\rm ref}}{\sqrt{U_{\rm lab}^2 + U_{\rm ref}^2}} \tag{6}$$

where *x* is the measured value by the participant laboratory, X_{ref} is the reference value (proposed by the organized laboratory), U_{lab} is the expanded uncertainty of *x*, and U_{ref} is the expanded uncertainty of X_{ref} . The data whose *En*-number satisfied $|En| \leq 1$ is judged as satisfactory performance. "|En| > 1" means unsatisfactory performance.

We found that some data reported from Lab.3 and Lab.4 shows |Z| > 3 and |En| > 1 in Figs. 9 and 10. In Fig 10b, there are many data reported from Lab.4



Fig. 9 Z-values of thermal diffusivity reported from each laboratory: (a) Armco iron and (b) isotropic graphite

which show unsatisfactory performance. The detector used by Lab.4 for the measurement of the temperature variation of the rear surface is a thermocouple (see Table 1). A fast response of the detector is needed for thermal-diffusivity measurements of IG-110 because the thermal diffusivity of the isotropic graphite is very high. It is therefore assumed that the behavior observed for results of Lab.4 is caused by the insufficient time response of the measurement system. Z-values and En-numbers are larger at 600 K and 900 K than for the other temperatures. This could be caused by the analysis method and the temperature measurement. Lab.4 analyzed temperature-rise curves using the half-time method, which assumes adiabatic experimental conditions. But above room temperature, it is often necessary to take into account the heat loss effect for the temperature-rise curve analysis. In addition, it is sometimes difficult to calibrate and measure accurately the temperature of the specimen in the case of an LF apparatus, because of the structure of the specimen holder, the temperature gradient in the specimen holder, and the stability of the test temperature which depends on furnace performance. On the other hand, the data at 1200 K show satisfactory performance, probably because the thermal diffusivity becomes small enough to avoid the problems due to the time response of the thermocouple, and because the temperature dependence of the thermal diffusivity become weak at high temperature. Moreover, the extrapolating method is sometimes difficult to apply to the results of Lab.4, as shown in Figs. 7 and 8.

Lab.3 had some difficulties to change the pulsed heating energy in order to apply the extrapolating procedure as shown in Figs. 7 and 8. Lab.3 also had difficulties to



Fig. 10 *En*-number of thermal diffusivity reported from each laboratory: (a) Armco iron and (b) isotropic graphite

evaluate the uncertainty of measurement. This could be the reasons why some data reported from Lab.3 show unsatisfactory performance.

Since we are able to infer the reasons for unsatisfactory performance, we determined Eq. 4 as the reference value of this comparison except for the outliers. Then we concluded that the results of the comparison with valid data agreed with each other, independent of the measurement system, measurement conditions, and analysis method of temperature-rise curve. We confirmed that the Si traceable and inherent thermal diffusivity can be determined and the extrapolating procedure is useful. And we recognized that there was some difficulty to realize these terms, dependent on the specifications of the measurement systems.

4 Conclusion

The first international comparison of LF thermal-diffusivity measurements organized within the BIPM frame was carried out between four participants in CCT-WG9 as CCT-P02. The pilot was NMIJ.

The thermal diffusivities of Armco iron and isotropic graphite were measured. The measurements were carried out for various thickness samples of each material by the LF method with an extrapolating procedure. The thermal-diffusivity data on Armco iron reported from the participants agree with the temperature dependence reported by LNE [18]. We used the equation estimated by LNE as the reference since the

measurements of the comparison were carried out at just four temperature levels. It was difficult to determine the temperature dependence of the thermal diffusivity of the Armco iron in the range from 300 K to 1200 K from just this study, because this material presents a magnetic phase transition in this temperature range. In the case of isotropic graphite, we determined the thermal diffusivity as a function of temperature using the measured data from this comparison.

We validated the results evaluating the deviations from the reference value, the Z-value, and the En-number. It was found that some data reported from Lab.3 and Lab.4 show large deviations, |Z| > 3 and |En| > 1. We supposed that these are caused by insufficient time response of the measurement system, the analysis method, and some difficulties in changing the pulsed heating energy for the extrapolating procedure. Since the reason for large deviations was known, we determined the reference values as the result of this comparison except for these outliers. Then we concluded that the results show fairly good agreement from room temperature to 1200 K among the participants in spite of their different measurement systems. It is also found that the inherent thermal diffusivity of the material is able to be determined independent of the specimen thickness and measurement conditions using the extrapolating procedure. We confirmed that the SI traceable and inherent thermal diffusivity can be determined and that the extrapolating procedure is useful. On the other hand, we recognized that there was some difficulty to realize these terms, depending on the specifications of the measurement systems. It is necessary to keep discussing and studying for establishment of a method to obtain the thermal diffusivity that is traceable to SI units and inherent by the LF method.

We considered the effect of the thermal expansion on thermal diffusivity. It was found that the thermal-expansion effect was very small and negligible in this case according to the calculation of thermal-expansion correction coefficients using the thermal-expansion coefficients of Armco iron and isotropic graphite measured by LNE.

We have a plan in the future to develop a metrology system for the thermophysical properties by CCT-WG9. For example, calibration and measurement capability (CMC) registration may be realized. We are discussing where we go as the next step on thermal-diffusivity measurements according to the results of this comparison in CCT-WG9.

References

- Bureau International des poids et Mesures, The International System of Units (SI), 8th edn. http:// www.bipm.org/utils/common/pdf/si_brochure_8.pdf. Accessed 16 May 2012.
- 2. http://www.bipm.org/en/home/. Accessed 16 May 2012
- 3. http://www.bipm.org/en/committees/cc/cct/. Accessed 16 May 2012
- 4. W.J. Parker, R.J. Jenkins, C.P. Butler, G.L. Abbott, J. Appl. Phys. 32, 1679 (1961)
- 5. M. Akoshima, T. Baba, Int. J. Thermophys. 26, 151 (2005)
- 6. B. Hay, J.R. Filtz, J. Hameury, L. Rongione, Int. J. Thermophys. 26, 883 (2005)
- NMIJ Certified Reference Materials Catalog 2011–2012, http://www.nmij.jp/service/C/ CRM_Catalogue20120315.pdf and http://www.nmij.jp/english/service/C/
- M. Akoshima, B. Hay, J. Zhang, L. Chapman, T. Baba, in *Proceedings of Thermal Conductivity 30/Ther*mal Expansion 18, ed. by D.S. Gaal, P.S. Gaal (DEStech Publications, Lancaster, 2010), p. 367–377
- 9. ISO/IEC Guide98:1995 "Guide to the expression of uncertainty in measurement" (GUM)

- M. Akoshima, T. Baba, in *Proceedings of Thermal Conductivity 28/Thermal Expansion 16*, ed. by R.B. Dinwiddie, M.A. White, L. McElroy (DEStech Publications, Lancaster, 2006), p. 497–506
- 11. A. Degiovanni, M. Laurent, Rev. Phys. Appl. 21, 229 (1986) [in French]
- 12. K. Shinzato, T. Baba, J. Therm. Anal. Calorim. 64, 413 (2001)
- 13. J.A. Cape, G.W. Lehman, J. Appl. Phys. 34, 1909 (1963)
- 14. D. Josell, J. Warren, A. Cezairliyan, J. Appl. Phys. 78, 6867 (1995)
- http://www.nmij.jp/~mprop-stats/thermophys/homepage/research/cfp32/index.html. Accessed 16 May 2012
- 16. T. Baba, A. Ono, Meas. Sci. Technol. 12, 2046 (2001)
- 17. J. Zhang, "Uncertainty_report_NIM" (for the comparison), private comunication
- B. Hay, J.-R. Filtz, J.-C. Batsale, Mesure de la diffusivité thermique par la méthode flash. Techniques de l'Ingénieur, R2955 (Paris, 2004) [in French]
- 19. ISO/IEC 17043:2010, "Conformity assessment-general requirements for proficiency testing"