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Thermal diffusivity measurement of BMS 10-102 thermal insulation material in a vacuum condition using a cyclic heating method

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ABSTRACT

With the many different types of spacecraft missions, insulation techniques are required to advance spacecraft thermal design. In order to create a reliable thermal design system, it is essential to characterize materials and basic properties accurately. In this paper, the cyclic heating method is used to measure the thermal diffusivity for a high-temperature porous material as an insulation material for a spacecraft. To verify this method, the thermal diffusivities of alumina (Al_2O_3) and polystyrene foam were measured at 47.5 °C (320.5 K) and 25.3 °C (298.3 K), respectively, in atmospheric conditions. The thermal diffusivity of the porous insulation material (BMS 10-102) was found to be 2.53×10^{-7} m^2/s at room temperature under atmospheric conditions. The thermal diffusivity of this porous insulation material was 8.88×10^{-8} m^2/s at room temperature under vacuum condition. The thermal diffusivities of porous insulation material under vacuum were reduced by 64.9% compared to the thermal diffusivities under the atmospheric condition. This difference is considered to be an effect of air in holes.

1. Introduction

With diversity in recent missions involving spacecrafts, new insulation techniques are required to advance the thermal design of these spacecrafts. Thermal diffusivities in a vacuum condition are required to apply composite materials or insulation materials into space structures. The use of a porous material is necessary to design lightweight, highly insulated structures.

There are a number of possible means of measuring thermal conductivity, each of them suitable for a limited range of materials, depending on the thermal properties and the temperature. Based on fundamental laws of heat transport, experimental methods for measuring the thermal conductivity of materials are divided into two groups: steady-state methods (e.g., the guarded hot plate method [1] and heat flow meter technique [2,3]) and transient methods (e.g., the laser flash method [4,5]). In general, the steady-state methods perform a measurement when the temperature of the material does not change with time. The thermal conductivity of a thermal insulation is usually measured using steady-state techniques. Such methods require large samples, long measurement times, large and complex measurement apparatus and well-engineered setups. The transient techniques, on the other hand, perform a measurement during the heating process. The advantage of such approaches is that measurements can be made relatively quickly and that only small samples are required. However, transient techniques are seldom used for the assessment of a thermal insulation material.

Transient thermal diffusivity measurements to determine the thermal conductivity of thermal insulation have been developed for many decades. First, the laser flash technique was modified by substituting step heating for flash heating [6]. The samples used in this method were relatively larger than the laser flash samples, but small compared to guarded hot-plate samples. Then, the three-point (3P) method [7] was developed. This method combined the advantages of rapid transient non-contact heating methods with the well-defined boundary conditions of steady-state methods. Thermal wave method that can be applied for a wide range of materials [8].

This paper presents the design of an apparatus for a cyclic heating method that is capable of measuring the thermal diffusivity of a material with low thermal diffusivity, such as a porous insulation material. BMS 10-102, which is widely used as an insulation material for space structures, is applied onto a carbon/epoxy composite body. This present method is a kind of thermal wave method for use in the thermal diffusivity measurement of thermal insulation. It requires three small samples (30 mm × 30 mm, 1–3 mm thickness), a short measurement time (about 1-h to take one thermal diffusivity), and relatively simple apparatus.

The thermal diffusivities of the BMS 10-102 material and carbon/epoxy composite were then measured under two sets of

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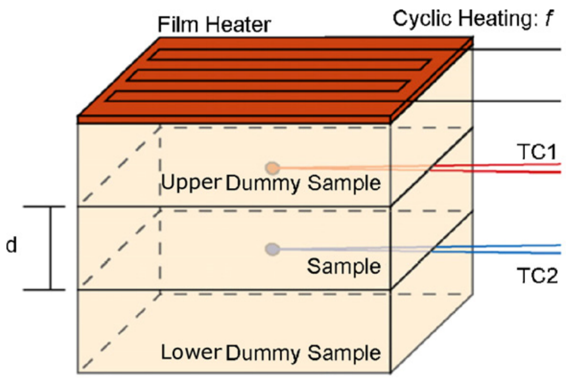

Fig. 1. Sample configuration.

conditions. The first condition is characterized by room temperature (298 K) under both atmospheric and vacuum conditions, and the second by a higher temperature (423 K) under atmospheric conditions.

2. Cyclic heating method

The cyclic heating method measures the thermal resistance, which is the phase difference of the thermal response from a heat source. Eq. (1) shows the calculation of the thermal diffusivity using the cyclic heating method. From this thermal diffusivity, the thermal conductivity is obtained with Eq. (2) [9]:

$$\alpha = \frac{\pi d^2 f}{\phi^2} \tag{1}$$

$$\lambda = \rho C_p \alpha \tag{2}$$

in which α is thermal diffusivity (m^2/s), d is specimen thickness (m), f is reply heat frequency (Hz), ϕ is phase difference (rad), λ is thermal conductivity (W m^{-1} K^{-1}), and ρC_p is the volumetric heat capacity (density times specific heat capacity; J m^{-3} K^{-1}).

2.1. Kunii's law

To evaluate the experiment results of the thermal diffusivity of a porous insulation material, here is applied the calculation method of Kunii's law is applied here as the following equations [10]:

$$\frac{\lambda_e}{\lambda_s} = 1 - e^{2/3} + e^{2/3}\left\{(1 - e^{1/3}) + \frac{e^{1/3}}{(\lambda_g/\lambda_s + (2/3)(h_r d_p/\lambda_s))}\right\}^{-1} \tag{3}$$

$$h_r = \frac{0.2268}{1 + e(1-\varepsilon)/\{2(1-e)\varepsilon\}}\left(\frac{T}{100}\right)^3 \tag{4}$$

where λ_e is thermal conductivity of a porous insulation (W m^{-1} K^{-1}), λ_s is thermal conductivity of solid (W m^{-1} K^{-1}), λ_g is thermal conductivity of gas (W m^{-1} K^{-1}), h_r is radiation heat transfer coefficient (W m^{-1} K^{-1}), d_p is diameter of hole (m), e is ratio of holes and ε is emissivity.

The ratio of holes, e, is calculated as following equation:

$$e = \frac{V_g}{V_0} = \frac{V_g}{V_s + V_g} \tag{5}$$

where V indicates volume, 0, g, and s indicate porosity, gas and solid, respectively.

3. Measurement method

3.1. Measurement of phase difference

The phase difference (ϕ) measures the thermal resistance of a sample, which is the difference of the thermal response from the sample heater (heat source). A detected signal contains a phase delay that occurs due to the thermal resistance and the response of the apparatus. To exclude this inherent phase delay, the specimens and a film heater were set as shown in Fig. 1. Three identical samples were stacked, and two thermocouples (TC1, TC2) were attached to the upper side and the lower side of the middle sample. The automated phase difference between the TC1 and TC2 calculation program was programmed using NI Labview 8.0.

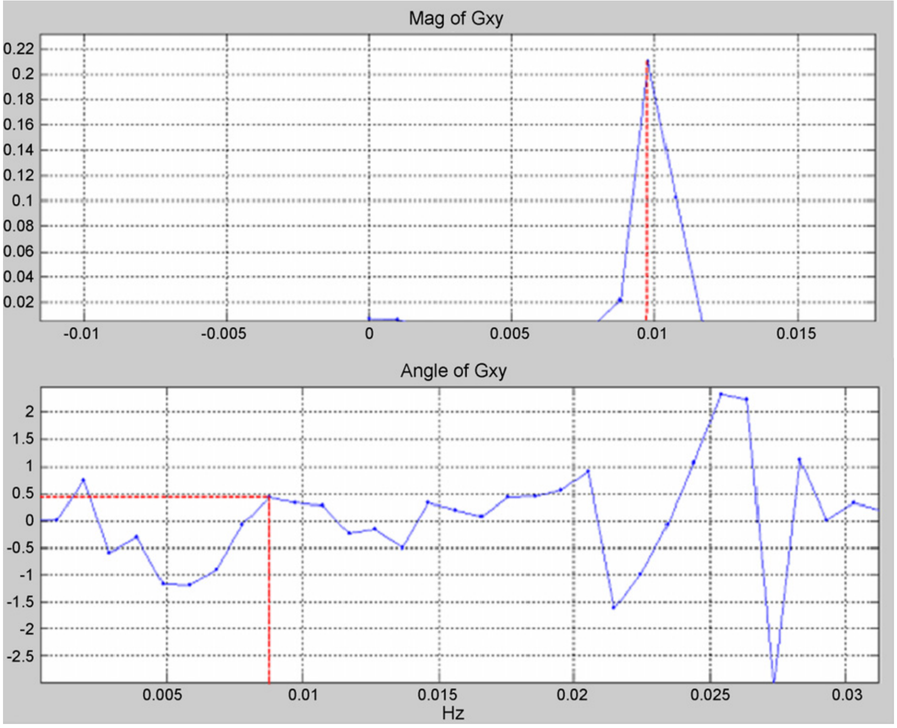

Fig. 2. How to get the unique phase differences ϕ_{s1} and ϕ_{s2}.

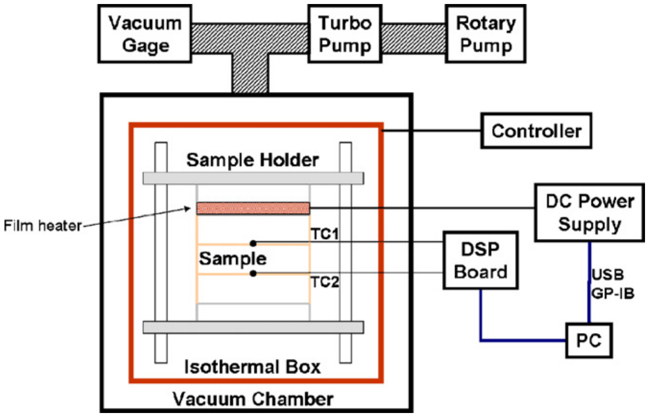

Fig. 3. Schematic diagram of the apparatus for the measurement of the thermal diffusivity.

The phase differences (ϕ_1, ϕ_2) and the detected signals are expressed by the following equations:

$$\phi_1 = \phi_A + \phi_{s1} \quad (6)$$

$$\phi_2 = \phi_A + \phi_{s2} \quad (7)$$

where ϕ_A is the phase delay inherent in the use of the apparatus, ϕ_{s1} is the unique phase differences of the upper dummy sample and ϕ_{s2} is the unique phase differences of the upper dummy sample with sample. The phase difference of the measurement sample ϕ is derived by subtracting Eq. (6) from Eq. (7). Fig. 2 shows how to get ϕ_{s1} and ϕ_{s2}. Dominant frequency is detected by FFT and then ϕ_{s1} and ϕ_{s2} can get by cross spectrum:

$$\phi = \phi_2 - \phi_1 = \phi_{s2} - \phi_{s1} \quad (8)$$

3.2. *Measurement apparatus*

Fig. 3 shows a schematic diagram of the apparatus used for the thermal diffusivity measurement by the cyclic heating method. The specimen and film heater (in Fig. 1) were put into an isothermal box, and the isothermal box was put into a test chamber. A DC power supply supplied voltage to the film heater, and this power supply was controlled by a PC. The communication between the DC power supply and the PC used a USB/GPIB connection. Signals from the thermocouples attached to the upper side and the lower side of the middle sample were obtained from a DSP board. The commercial program NI Labview 8.0 was used to control the voltage supply of the film heater. This software also provided data from the thermocouples. The test chamber isolated the test apparatus from the outer environment.

The isothermal box system as shown in Fig. 4 is the apparatus used to elevate and maintain the surrounding temperature. It can control the surrounding temperature in a range from room temperature to 573 K within ±0.01 K of accuracy.

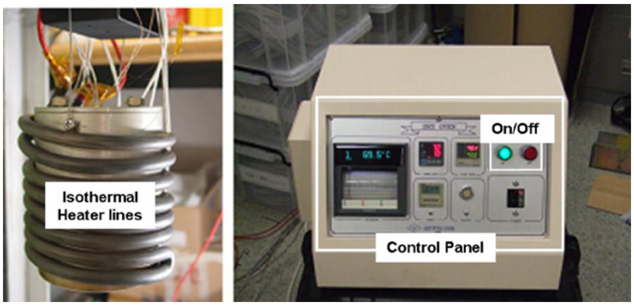

Fig. 4. Isothermal box and its controller.

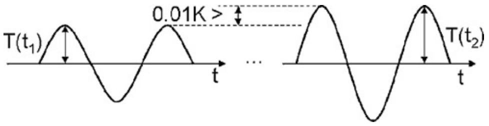

Fig. 5. Criterion for the measurement of the specimen temperature.

Table 1
Characteristics of the specimen, alumina (Al_2O_3) [11].

	Value
Density	3.92 g/cm^3
Hardness	1600 HV 10
	Component (wt%)
Purity	$Al_2O_3 > 99.7$ $MgO < 0.1$ $SiO_2 < 0.1$ $Na_2O < 0.05$ $Fe_2O_3 < 0.02$

Table 2
Comparison of the measurement value and a reference value.

Temperature	320.5 K
Measurement value	9.7×10^{-6} m^2/s
Reference value	9.1×10^{-6} m^2/s
Deviation	6.6%

Using this system, a criterion for the specimen temperature measurements could be controlled. The criterion can be expressed by the formula in Eq. (9) and shown in Fig. 5:

$$|T(t_1) - T(t_2)| < 0.01\,\text{K} \quad (9)$$

Here, $T(t_1)$ and $T(t_2)$ are the peak temperatures at the time of t_1 and t_2, respectively. One phase difference value is calculated from five cycles of temperature. To obtain one thermal diffusivity value, fifteen phase difference values are averaged.

3.3. *Verification of the measurement system*

3.3.1. *Alumina (Al_2O_3)*

To verify the cyclic heating method, alumina (Al_2O_3) was tested. This material has a well-known thermal diffusivity as a function of temperature. The thermal diffusivity of alumina was measured under at 320.5 K (47.5 °C) under atmospheric conditions. The specimen thickness was 5.7 mm.

Eq. (10) is the thermal diffusivity of alumina as a function of the temperature [11]:

$$\alpha = \frac{3.86 \times 10^{-3}}{T - 87.6} - 1.49 \times 10^{-5} + 3.08 \times 10^{-8} T - 2.65 \times 10^{-11} T^2 + 8.27 \times 10^{-15} T^3 \quad (10)$$

Here, α (m^2/s) is thermal diffusivity, and T (K) is temperature.

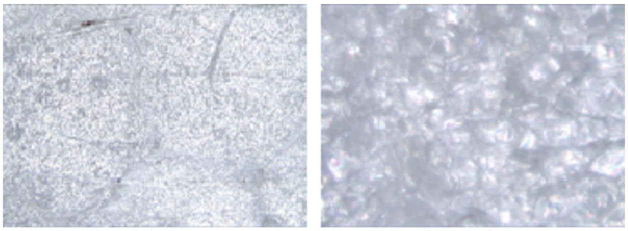
(a) 50times (b) 350times

Fig. 6. LM images of a cross-section of the polystyrene foam.

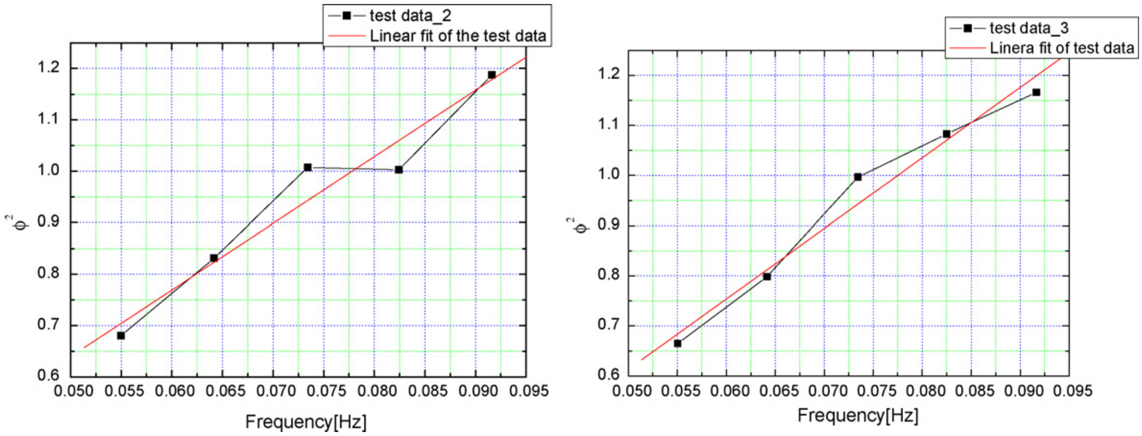

Fig. 7. Linear fit of frequency vs. square of the phase difference.

The characteristics of the alumina are shown in Table 1.

The thermal diffusivity was calculated from Eq. (10) as a reference value. Table 2 shows the measurement value and the reference value. The percent deviation between the value after using the cyclic heating method and the reference value was 6.6%.

3.4. Polystyrene foam

The thermal diffusivities of polystyrene foam were measured to verify this system for porous materials. Polystyrene foam is typically used as a general purpose insulation material in industrial areas. Additionally, it is characterized by low thermal conductivity and low density. The sizes of these specimens were 25 mm × 25 mm × 1.85 mm. Fig. 6 shows light microscope images of a cross-section of the polystyrene foam.

For the cyclic heating method, it was crucial to determine the voltage level and frequency of the DC power supply. These values vary according to the material and specimen thickness. At a specific temperature, the thermal diffusivity of a material should be constant. Therefore, from Eq. (1), the ratio of the frequency and square of the phase difference should have a uniform value; specifically it should have a linear relationship.

Fig. 7 shows the two linear fits of frequency versus the square of the phase at room temperature (298 K) under atmospheric conditions. The two equations of the relationship are shown in Eqs. (10) and (11):

$$\phi^2 = 12.95179f - 0.00793 \tag{10}$$

$$\phi^2 = 14.06102f - 0.08981 \tag{11}$$

Table 3 shows the deviation of the thermal diffusivities from those of the reference material. The reference value of polystyrene foam was contributed by a testing center in Japan. The results obtain by the present measurement system agreed within approximately 10% of deviation from the reference values.

Fig. 8 shows atmospheric and vacuum thermal diffusivity data compared with Japan testing center values. In this case, atmospheric data is the average value of (1) and (2) from Table 3. In the vacuum condition (3.40×10^{-5} Torr), the thermal diffusivity of polystyrene foam was 2.95×10^{-7}. This is 62.5% lower than its thermal diffusivity in atmospheric conditions.

Table 3
Deviations of thermal diffusivities from the reference value.

	Temperature	Thickness (mm)	Thermal diffusivity (m^2/s)	Deviation (%)
Reference value	298.0 K	–	7.38×10^{-7}	–
(1)	298.3 K	1.85	8.31×10^{-7}	12.3
(2)	298.5 K	1.85	7.70×10^{-7}	4.3

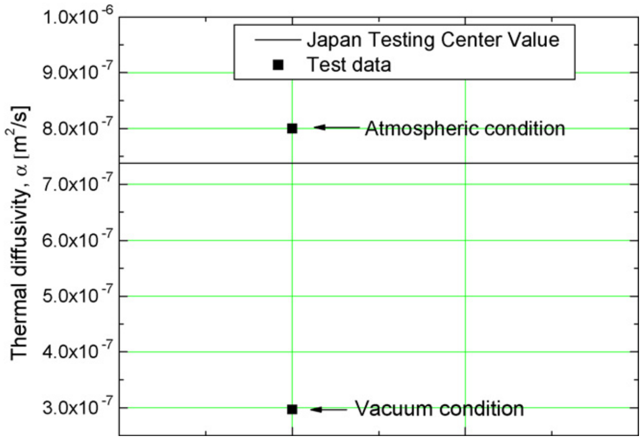

Fig. 8. Thermal diffusivities of polystyrene foam.

4. Thermal diffusivities of BMS 10-102

4.1. Thermal diffusivities

BMS 10-102, which meets the requirements for a Type I, Class I, Grade 25 material, is a high temperature-resistant insulation coating material. BMS 10-102 was tested under three types of conditions.

Table 4 and Fig. 9 show the thermal diffusivities of BMS 10-102 with minimum and maximum values and calculated values by

Table 4
Thermal diffusivities of BMS 10-102 under three conditions.

	Atmospheric condition	Vacuum condition (7.5×10^{-5} Torr)
298 K	Max. 2.60×10^{-7} m^2/s Min. 2.47×10^{-7} m^2/s Avg. 2.52×10^{-7} m^2/s Kunii's law: 2.51×10^{-7} m^2/s	Max. 8.96×10^{-8} m^2/s Min. 8.74×10^{-8} m^2/s Avg. 8.88×10^{-8} m^2/s Kunii's law: 1.32×10^{-7} m^2/s
423 K	Max. 2.60×10^{-7} m^2/s Min. 2.42×10^{-7} m^2/s Avg. 2.50×10^{-7} m^2/s Kunii's law: 2.51×10^{-7} m^2/s	

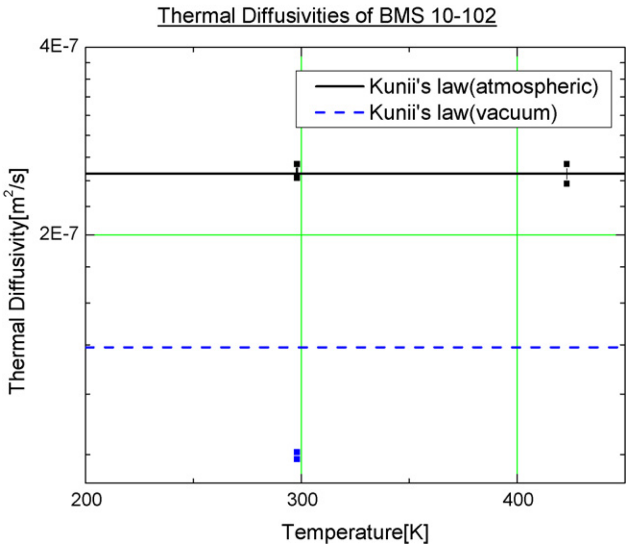

Fig. 9. Thermal diffusivities of BMS 10-102.

Kunii's law. λ_g was assumed to be approximately zero for the vacuum condition in Eq. (3). The value of Kunii's law was 49.2% higher than test result for the vacuum condition.

Only a slight, insignificant difference is shown between the thermal diffusivity measurements at the atmospheric room temperature condition and at the atmospheric 423 K conditions. The thermal diffusivity at room temperature under a high vacuum was reduced by 64.6% compared to the thermal diffusivity at the atmospheric room temperature condition. The test results under the atmospheric condition are in good agreement with the values of Kunii's law, whereas the result under the vacuum condition is not. From this comparison, Kunii's law is not suitable for a vacuum condition. Therefore, experiments are required to obtain the thermal diffusivity data under a vacuum condition. The material properties for Kunii's law in Eqs. (3) and (4) are shown in Table 5. In Table 5, λ_s (thermal conductivity of a solid) is assumed as the thermal conductivity of silicon, e (ratio of holes) is obtained by image processing and ε (emissivity) is an assumed value. Only a slight, insignificant difference is shown between the thermal diffusivity measurements at the atmospheric room temperature condition and at the atmospheric 423 K conditions.

4.2. Uncertainty

Sample thickness, thermocouple positions, applied frequency, voltages delivered by the thermocouple wires, and roughness of the sample surface, etc., can cause uncertainty in measurement accuracy. The uncertainty of the cyclic heating method was determined with the ISO Guide [12]. In this paper, applied frequency, sample thickness, phase difference and temperature were considered as uncertainty components. Tables 6–8 show uncertainty components and values under each three conditions. Also the estimated relative expanded uncertainty (coverage factor k=2) of the thermal diffusivity $U(\alpha)$ is shown in each table.

Table 5
Material properties for Kunii's law.

Parameter	Value
ρ (kg/m^3)	313.2
e	0.6749
ε	0.1
d_p (m)	5×10^{-5}
C_p (J kg^{-1} K^{-1})	1256
λ_s (W m^{-1} K^{-1})	0.298

Table 6
Uncertainty components under atmospheric condition (298 K).

Uncertainty components	Atmospheric condition (298 K)	
	Sensitivity coefficient	Standard uncertainty
Frequency	5.0930×10^{-6} m^2	6.0151×10^{-9} Hz^2
Phase difference	-1.2969×10^{-7} m^2/s	1.8984×10^{-2} rad^2
Sample thickness	1.0186×10^{-4} m^2/s	8.3333×10^{-10} m^2
Temperature	6.0000×10^{-13} m^2/s K	2.9485×10^{-3} K^2
Combined standard uncertainty, $u_c(\alpha)$	1.8114×10^{-8} m^2/s	
Expanded standard uncertainty, $U(\alpha)$ $(k=2)$	3.6228×10^{-8} m^2/s	

Table 7
Uncertainty components under atmospheric condition (423 K).

Uncertainty components	Atmospheric condition (423 K)	
	Sensitivity coefficient	Standard uncertainty
Frequency	1.8317×10^{-6} m^2	4.2226×10^{-9} Hz^2
Phase difference	-1.0754×10^{-7} m^2/s	5.3491×10^{-3} rad^2
Sample thickness	1.1327×10^{-4} m^2/s	8.3333×10^{-10} m^2
Temperature	1.2×10^{-12} m^2/s K	9.472×10^{-3} K^2
Combined standard uncertainty, $u_c(\alpha)$	8.5187×10^{-9} m^2/s	
Expanded standard uncertainty, $U(\alpha)$ $(k=2)$	1.7038×10^{-8} m^2/s	

Table 8
Uncertainty components under vacuum condition (298 K).

Uncertainty components	Vacuum condition (298 K)	
	Sensitivity coefficient	Standard uncertainty
Frequency	2.5155×10^{-6} m^2	6.4141×10^{-9} Hz^2
Phase difference	-2.9161×10^{-8} m^2/s	3.4149×10^{-3} rad^2
Sample thickness	3.0332×10^{-5} m/s	8.3333×10^{-10} m^2
Temperature	6.0×10^{-13} m^2/s K	1.4814×10^{-1} K^2
Combined standard uncertainty, $u_c(\alpha)$	1.9264×10^{-9} m^2/s	
Expanded standard uncertainty, $U(\alpha)$ $(k=2)$	3.8528×10^{-9} m^2/s	

5. Conclusions

To measure the thermal diffusivities of a porous insulation material, a measurement technique using a cyclic heating method was developed. To verify the measurement technique, the isothermal box system that was able to control the surrounding temperature in a range from room temperature to 573 K within ±0.1 K of accuracy was created. To verify the effect of the air in holes of the porous insulation material, a vacuum system was applied to the isothermal box system. A phase difference criterion of the porous materials for cyclic heating method was proposed. This system is also able to measure the values of α and ε for the same specimen. The measurement method was verified by measuring the thermal diffusivity of an alumina (Al_2O_3) specimen. The test results of the porous materials are described below.

For polystyrene foam, which was selected as a standard material from among the many types of porous materials, the thermal diffusivities under the atmospheric room temperature condition agree within approximately 10% of deviation from the reference value. In the vacuum condition (3.40×10^{-5} Torr), the thermal diffusivity of the polystyrene foam was 2.95×10^{-7} m^2/s. This is 62.5% lower than the thermal diffusivity of the polystyrene foam in the atmospheric condition.

For a porous insulation material (BMS 10-102) which is one of the insulation materials used in aerospace structures, the thermal diffusivity at room temperature under a high vacuum was reduced by 64.6% compared to the thermal diffusivity under atmospheric room temperature conditions. Hence, it is concluded that under a vacuum condition, heat transfer by convection through pores is removed. There was essentially no difference in the thermal diffusivity results at the atmospheric room temperature condition and at 423 K under atmospheric conditions. From this result, it is expected that the thermal diffusivity of an insulation material in the temperature range from 298 K to 423 K is rarely influenced by the temperature. In addition to this research, the calculation method of porous insulation is needed to develop in vacuum condition.

Acknowledgement

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