# THERMAL CONDUCTIVITY MEASUREMENT OF THERMAL INSULATION UNDER HIGH TEMPERATURE

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## Abstract

*We have newly developed an apparatus for measuring thermal conductivity of thermal insulations in the temperature range from 100 to 1500 ℃ under both atmospheric pressure and vacuum conditions by applying the cyclic heat method. For the purpose of investigating the accuracy of measurement of the apparatus, thermal conductivities of fibrous insulations were measured, and the results were compared with those obtained by other apparatuses which are based on the guarded hot plate method and the cyclic heat method. From the comparison, the measured values were confirmed to agree with each other within ±10 % in the temperature range from 100 to 1000 ℃. Also, the present apparatus is shown to be applicable for measuring thermal conductivities of the two kinds of specimens simultaneously. Alumina silica board and alumina silica fiber were measured simultaneously in the temperature range from 200 to 800 ℃. The results agreed well with those obtained by one specimen method. Further, we have tried to measure the thermal conductivity of Alumina silica board under vacuum condition of about 1 Pa at high temperature. The obtained values under vacuum condition were shown to be less than those obtained by subtracting thermal conductivity of air from those of the specimen under atmospheric pressure. Also, for the purpose of investigating degradation of thermocouple, the changes in electromotive force of the thermocouple with time were measured by exposing it to high temperature environment maintained at 1500 ℃.*

## 1 Introduction

Thermal insulations are widely used in many industries, including space, nuclear power plant, and electric power industries. Recently, with increase in requirement for using those thermal insulations at higher temperatures and in vacuum conditions, it has been becoming more and more important to know the thermal conductivities under such severe conditions. Especially, vacuum insulations have become of major interest lately, because the thermal conductivity becomes smaller with decrease in the pressure inside of the insulations.

On the other hand, concerning the measurement method of thermal conductivity, the guarded hot plate (GHP) method, which is one of the representative steady state methods, is generally used for this kind of measurement. However, there are few reports about the thermal conductivities of thermal insulations at higher temperatures over 1000 ℃, because it is difficult to make measurements under such higher temperature conditions.

Further, so far as we know, although unsteady state methods like the cyclic heat method are applicable to the thermal conductivity measurement under the higher temperature and vacuum conditions, there are few reports by applying these methods.

To cope with issues mentioned above, we have newly developed an apparatus for measuring thermal conductivities of thermal insulations in the temperature range from 100 to 1500 ℃ and both in the atmospheric pressure and vacuum conditions by applying the cyclic heat method [1]. In this paper, the measured results of the thermal conductivity of thermal insulations under high temperature and vacuum conditions are shown.

## 2 Principle of Measurement

As is well known, the cyclic heat method provides two kinds of ways for thermal diffusivity measurement; one is a way to measure the phase lag of generated temperature wave which propagates through a specimen, the other is a way to measure the amplitude decay of the temperature wave. Anyhow the physical value obtained from these two ways is the thermal diffusivity of a specimen, so the thermal conductivity of the specimen is obtained by multiplying the specific heat and the bulk density by the thermal diffusivity.

Figure 1 shows a schematic of the temperature wave propagating through the specimen. In the figure, the symbols $\theta$ and $t$ represent the temperature and the time, respectively. Here, the $x$-axis is taken along the direction of the thickness in the specimen.

On referring to the figure, the time lag $\phi$ and the amplitude decay $A$ of the temperature

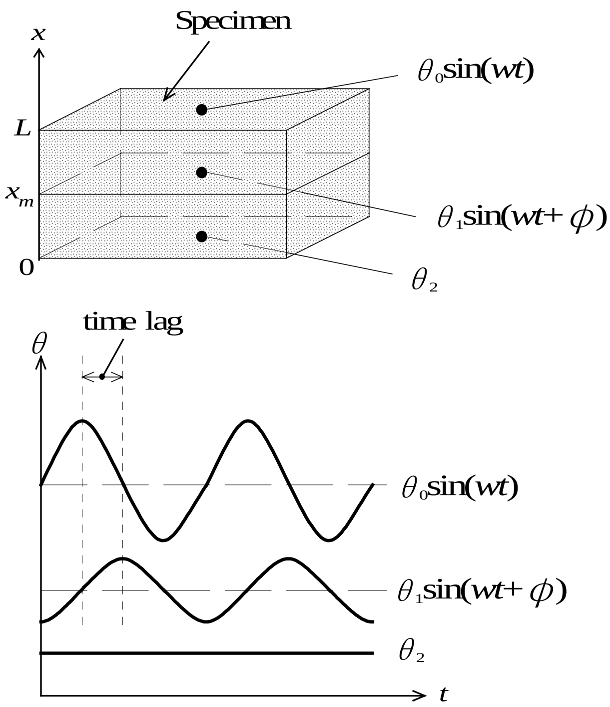


Fig. 1 Schematic of temperature wave

wave are expressed by the following equations [1].

$$\phi = \arg\left\{\frac{\sinh kx_m(1+i)}{\sinh kL(1+i)}\right\} \tag{1}$$

$$A = \sqrt{\frac{\cosh 2kx_m - \cos 2kx_m}{\cosh 2kL - \cos 2kL}} \tag{2}$$

where $k$ is the attenuation coefficient defined by the following Eq. (3), $x_m$ is the measured point of the temperature in the specimen, $L$ is the thickness of the specimen, and $i$ is the imaginary unit.

$$k = \sqrt{\frac{\omega}{2\kappa}} \tag{3}$$

Here, $\kappa$ is thermal diffusivity, and the angular frequency $\omega$ included in the above equation is related to the heating period $T$ through the following Eq. (4).

$$\omega = \frac{2\pi}{T} \tag{4}$$

Therefore, the required thermal conductivity $\lambda$ of the specimen is obtained by

$$\lambda = \rho c \kappa \tag{5}$$

where $\rho$ and $c$ are the bulk density and the specific heat of the specimen, respectively.

## 3 Measuring Apparatus

The present apparatus newly developed can measure the thermal conductivity of not only one kind of specimen but also even two kinds simultaneously.

### 3.1 In the Case of One Kind of Specimen

Figure 2 shows a schematic of the apparatus. Classifying roughly, the apparatus is composed of a measuring cell and a control system.

The measuring cell is made up of a pair of specimens and four heaters; a cyclic heater, a lower heater, an upper heater, and a cylindrical heater. An arbitrary temperature wave is generated by a combination of the cyclic heater and a function generator. The upper heater plays a role of transmitting the temperature wave effectively to the specimen, and the lower heater acts as an absorber of the incoming temperature

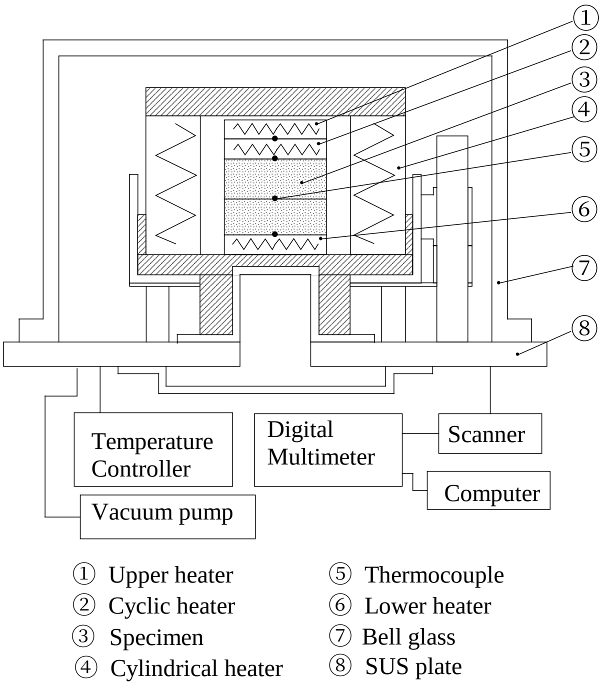


Fig. 2 Schematic of measuring apparatus
(In the case of one kind of specimen)

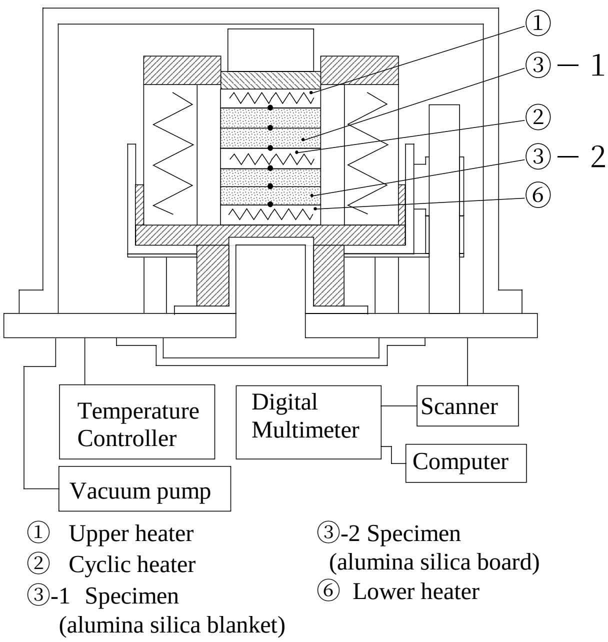


Fig. 3 Schematic of measuring apparatus
(In the case of two kinds of specimens)

wave. A pair of specimens, each of which is a rectangular prism in shape and measures 120 mm in length, 120mm in width and about 25 mm in thickness, is placed between the cyclic heater and the lower heater. Concerning temperature measurement, R-type thermocouple of 0.3 mm in diameter is inserted between the two specimens.

The control system is composed of a temperature controller, a function generator, a digital multimeter, a power supply, and a personal computer. The vacuum pump, which is a rotary type, makes arbitrary degree of vacuum conditions inside of the bell glass.

The specific heat of the specimen was measured by the drop calorimeter of our own making [2, 3].

### 3.3 In the Case of Two Kinds of Specimens

Figure 3 shows a schematic of the apparatus. In this measurement, one pair of specimens is placed between the cyclic heater and the lower heater (lower specimen pair); the other pair of specimens is placed between the cyclic heater and the upper heater (upper specimen pair). Then, two R-type thermocouples are inserted between the lower side and upper side surfaces of the cyclic heater, respectively.

The temperature waves, which are generated by the cyclic heater, propagate in the two directions; upper and lower specimen pairs. Therefore, the thermal conductivities of two kinds of specimens are obtained simultaneously by measuring the time lags or the amplitude decays in the specimens.

## 4 Results and Discussion

### 4.1 Effect of Heating Period on Thermal Conductivity Measurement

Figure 4 shows the effect of the heating period $T$ on thermal conductivity measurement of alumina silica board (bulk density: 400 kg/m$^3$) in the atmospheric pressure. The open diamond, open square, open triangle, and open circle show the measured thermal conductivities $\lambda$ at temperatures $\theta$ = 200, 600, 1000, 1500 °C, respectively. On the other hand, the solid, broken, dash-dotted, and two-dot chain lines are

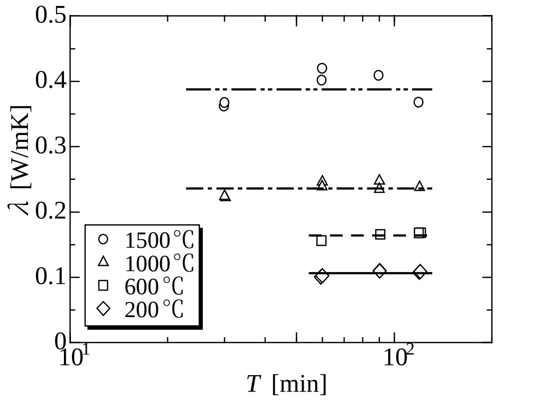


Fig. 4 Effect of heating period on thermal conductivity measurement of alumina silica board

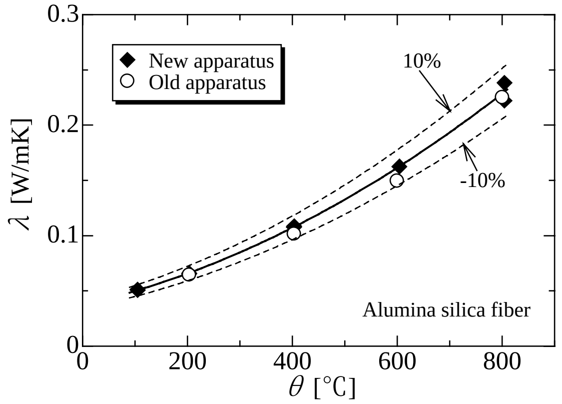


Fig. 6 Comparison of new apparatus with old apparatus

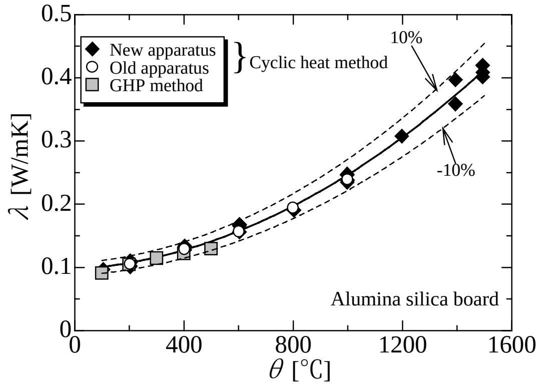


Fig. 5 Comparison of new apparatus with old apparatus and GHP method

the mean values at each temperature averaged over the measured heating period *T*.

From the figure, it seems that the measured thermal conductivities $\lambda$ at each temperature are not affected by the heating period *T* over 20 to 120 minutes.

## 4.2 Comparison with Other Methods

Figure 5 shows the thermal conductivity $\lambda$ of alumina silica board (bulk density: 400 kg/m$^3$) measured by using the present apparatus in the temperature range from 100 to 1500 °C under the atmospheric pressure. In the figure, measured results by the old apparatus [4] and the GHP method (EKO Corp.; HC-090) are also shown for reference. The closed diamond, open circle, and closed square denote the measured values using the present apparatus, the old apparatus, and the GHP method, respectively. The solid line is the fitted curve by the least squares method of thermal conductivities measured by using the present apparatus. The broken lines are ±10 % deviations from the solid line. It can be seen that the results obtained by the present apparatus agree well with those measured by the old apparatus and the GHP method.

In the same way, the thermal conductivity $\lambda$ of alumina silica fiber (bulk density: 150 kg/m$^3$) measured by using the present apparatus and the old apparatus in the temperature range from 100 to 800 °C is shown in Fig. 6. The meanings of the closed diamond, open circle, and the solid and broken lines are the same as those in Fig. 5. In this case, too, the results obtained by the present apparatus agree well with those measured by the old apparatus.

## 4.3 Simultaneous Measurement of Thermal Conductivities of Two Kinds of Specimens

Figure 7 shows the measured thermal conductivities $\lambda$ of alumina silica board (bulk density: 400 kg/m$^3$), which is placed between the cyclic heater and the lower heater, and alumina silica fiber (bulk density: 150 kg/m$^3$), which is placed between the cyclic heater and the upper heater, obtained simultaneously by using the present apparatus in the temperature range from 200 to 800 °C under atmospheric pressure. The open and closed circles show the

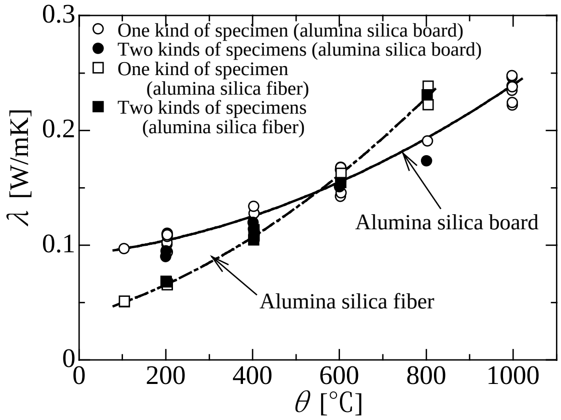


Fig. 7 Thermal conductivities obtained by simultaneous measurement

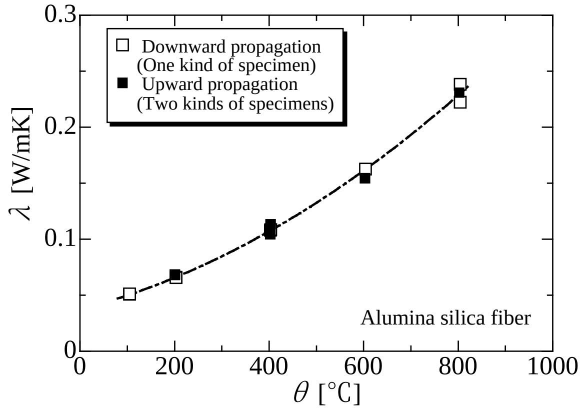


Fig. 8 Comparison of upward propagation with downward propagation of temperature wave for alumina silica fiber

results of alumina silica board measured by using the apparatuses shown in Figs. 2 and 3. The open and closed squares also show the results of alumina silica fiber. Here, the solid and dash-dotted lines drawn in the figure denote the fitted curves by the least square method of thermal conductivities of the one kind of specimen. From the figure, it can be seen that the simultaneously measured values agree fairly well with those obtained by one specimen method.

It was considered first that one temperature wave was possibly interfered with the other in the simultaneous measurements. However, the measured results showed that the present simultaneous measurement was independent of the interference of the two temperature waves. One of the reasons considered here is that, in the present measurements, the thermal conductivities of both specimens are fairly low. Namely, both specimens have the high attenuation coefficients *k*.

The attenuation coefficient is expressed by the following equation from Eqs. (3), (4), and (5).

$$k = \sqrt{\frac{\pi \rho c}{T \lambda}} \qquad (6)$$

Therefore, the lower the thermal conductivity $\lambda$ is, the larger the attenuation coefficient $k$ becomes. So, in the present measurements, it is considered that the temperature waves are attenuated enough to neglect the interference of each waves.

However, if either specimen (or both specimens) has high thermal conductivity, the two temperature waves will interfere with each other. This is because the temperature wave propagating through the specimen with higher thermal conductivity could be reflected from the surface whose temperature is maintained constant. In such a case, the measurement will be affected by the superposition of two temperature waves. To cope with the issue, the heating period $T$ will need to be shortened enough for the attenuation coefficient to be large.

In Fig. 7, the measured results of the alumina silica fiber show that the measured values on the upper side, in which the temperature wave is propagating upward through the specimen, agree with the measured values on the lower side, in which the temperature wave is propagating downward through the specimen. To facilitate comparison, the measured results of the alumina silica fiber are shown in Fig. 8. Although the measured values on the upper side seem to be affected by the natural convection in the vacant space of the specimen, the result shows that the measurement on the upper side is independent of the natural convection. The reason was considered that the vacant space was too small to generate the natural convection.

In the case of fibrous insulation, the generation of the natural convection is generally examined by the modified Rayleigh number *Ra*, which is expressed by the following equation [5].

$$Ra = \frac{g\beta\Delta\theta L^3}{\nu\kappa_a}\frac{h}{L^2} \qquad (7)$$

where *g* is the gravitational acceleration, $\beta$ is the volume coefficient of expansion, $\Delta\theta$ is the temperature difference, ν is the kinematic viscosity, $\kappa_a$ is the thermal diffusivity of the fluid, *L* is the thickness of the specimen, and *h* is the permeability defined by the following equation [6].

$$h = \frac{r^2\delta^3}{122(1-\delta)^2} \qquad (8)$$

where *r* is the diameter of a fiber, and $\delta$ is the porosity of the specimen. Namely, when *Ra* is larger than the critical modified Rayleigh number $Ra_{cr}$ ( = $4\pi^2$), the natural convection is induced in the fibrous insulation.

For example, when the mean temperature of the specimen is 500 °C, the bulk density $\rho$ is 150 kg/m$^3$, the true density is 2900 kg/m$^3$, the diameter of the fiber is 4 x 10$^{-6}$ m, the thickness of the specimen *L* is 0.04 m, the volume coefficient of expansion $\beta$ is 1/773 K$^{-1}$, the kinematic viscosity ν is 7.63 x 10$^{-5}$ m$^2$/s, the thermal diffusivity of the air $\kappa_a$ is 1.06 x 10$^{-4}$ m$^2$/s [7], and the temperature difference $\Delta\theta$ is 30 °C, the permeability *h* is

$$h = \frac{(4\times10^{-6})^2\times0.95^3}{122\times0.05^2} = 4.5\times10^{-11} \qquad (9)$$

Therefore, *Ra* is

$$Ra = \frac{9.8\times\frac{30}{773}\times0.04\times4.5\times10^{-11}}{7.63\times10^{-5}\times1.06\times10^{-4}} = 8.5\times10^{-5} \qquad (10)$$

Since *Ra* is less than $Ra_{cr}$ (=39.5), the natural convection isn't induced in the vacant space of the specimen. Therefore, the present measurements on the upper side are independent of the natural convection.

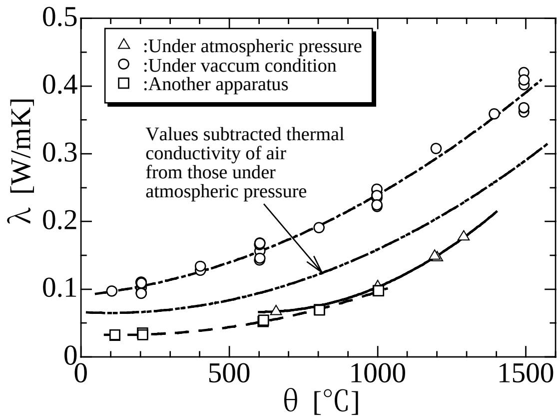


Fig. 9 Comparison of values under atmospheric pressure with those under vacuum condition

## 4.4 Thermal conductivity of thermal insulation under vacuum condition

Figure 9 shows the thermal conductivities of Alumina silica board under vacuum condition of about 1 Pa. The specimen was placed between the upper heater and the cyclic heater in the measurement. In the figure, measured results under atmospheric pressure are also shown for reference. The open circle and open triangle denote the measured values under atmospheric pressure and vacuum conditions, respectively. The solid and dash-dotted lines are the fitted curves by the least squares method of thermal conductivities measured under atmospheric pressure and vacuum conditions, respectively. Further, the two - dot chain line shows the results obtained by subtracting thermal conductivities of air from the thermal conductivities under atmospheric pressure.

From this figure, it is obvious that the results measured under vacuum condition are less than those obtained by subtracting thermal conductivity of air from those of the specimen under atmospheric pressure. By way of precaution, the same kind of specimen was measured using the old apparatus, which was based on the cyclic heat method. The results are shown by open squares and the broken line, which are the fitted curves by the least squares method. These values agreed with those measured by the new apparatus.

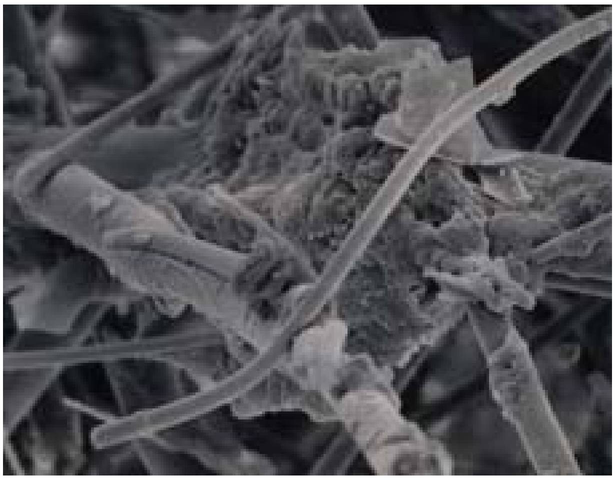

Fig. 10 SEM image of the specimen (Junctions between fibers each other or between particle and fiber; ×2000)

The reason seems that porosity in the inside of junctions between fibers each other or between particle and fiber had many small vacuum condition spaces. Figure 10 shows a SEM image of the junctions in the specimen. If the whole thermal resistance of the specimen was composed of thermal equivalent parallel circuit based on conductions in solid, radiation, and gas, the measured thermal conductivity under the vacuum condition would agree with the subtracted thermal conductivity. However, in the case of the present specimen shown in Fig. 10, the thermal resistance seems to be complex by concluding the vacuum porosity in the inside of junctions between fibers each other, in which heat conducts through the solid as media.

### 4.5 Degradation of Thermocouples

In the present measuring apparatus, thermocouples are exposed to quite high temperature environment. Although R-type thermocouples, which are durable at high temperature, are used in everywhere in the apparatus, some thermocouples which are exposed at temperature over 1000 ℃ are considered to be corroded. For the purpose of investigating degradation of thermocouple, the surfaces of the thermocouple before and after exposures were observed directly by a scanning electron microscope (SEM). Also the changes in electromotive force of the thermocouple with

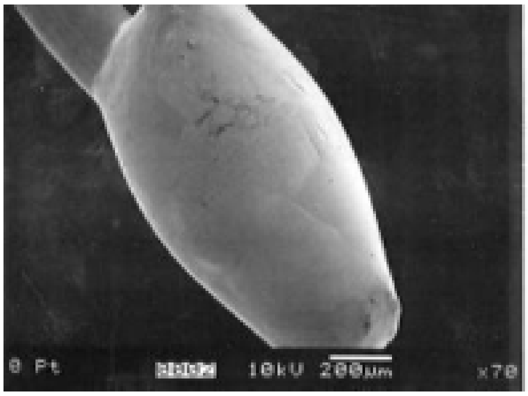


Fig. 11 SEM image of hot junction surface of R-type thermocouple before being used

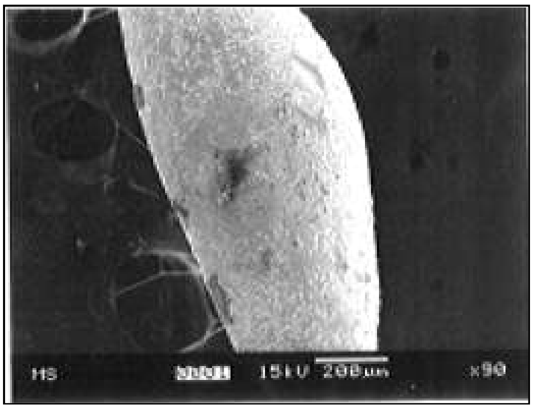


Fig. 12 Similar SEM image after being used for 42 hours at 1500 °C

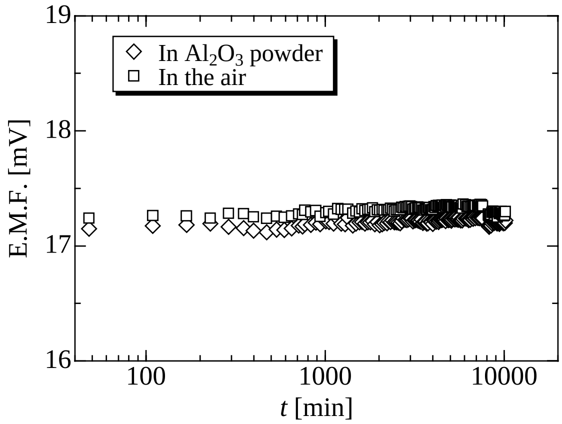


Fig. 13 Aged deterioration of electromotive force of thermocouples kept at 1500 °C

time were measured by exposing it to high temperature environment maintained at 1500℃.

Figure 11 shows a SEM image of hot junction surface of R-type thermocouple before being used. On the other hand, Fig. 12 shows the similar SEM image after being used for 42 hours at 1500 °C. Compared with the SEM image shown in Fig. 11, the hot junction surface shown in Fig. 12 exhibits a rough state with many uneven spots. This means the thermocouple could be corroded by penetration of $Al_2O_3$ and $SiO_2$ from the specimen.

Figure 13 shows measured electromotive force changes of thermocouples placed in the $Al_2O_3$ powder and in the high temperature air of furnace, both of which were kept at 1500 ℃ for 167 hours. As seen from the figure, the electromotive force of the thermocouple hardly changes even after 167 hours of the present endurance test. However, these thermocouples were broken after that, about 170 hours. Therefore, according to our experiences, thermocouples exposed to this kind of environment are recommended to be checked and changed regularly.

## 5 Conclusions

The thermal conductivities of alumina silica board and alumina silica fiber have been measured in the temperature range from 100 to 1500 °C under atmospheric pressure and vacuum conditions by using newly developed apparatus which applied the cyclic heat method. Also, the present new apparatus is shown to measure the thermal conductivities of the two kinds of specimens simultaneously.

Also, we have tried to measure the thermal conductivity of Alumina silica board under vacuum condition of about 1 Pa at high temperature. The obtained values under vacuum condition were shown to be less than those obtained by subtracting thermal conductivity of air from those of the specimen under atmospheric pressure.

Further, an example of hot junction corrosion of thermocouples by $Al_2O_3$ and $SiO_2$ was shown. Thermocouples exposed at the high temperature condition were broken by the degradation.

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