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DEVELOPMENT OF HIGH TIME-RESOLUTION LASER FLASH EQUIPMENT FOR THERMAL DIFFUSIVITY MEASUREMENTS USING MINIATURE-SIZE SPECIMENS

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ABSTRACT

For measurements of thermal diffusivity of miniature-size specimens heavily irradiated by neutrons, a new Q-switched laser-flash instrument was developed. In the present instrument the time resolution was improved to 0.1 ms by using a laser-pulse width of 25 ns. The realization of high time-resolution made it possible to measure the thermal diffusivity of thin specimens. It is expected that copper of 0.7mm thick, and SUS 304 of 0.1mm could be used for the measurements. In case of ATJ graphite, 0.5mm thick specimen could be used for the reliable measurement in the temperature range of 300-1300K.

Key words; Experimental Techniques, Thermophysical Properties.

1. Introduction

Thermal property changes due to heavy irradiations should be addressed extensively, for a reliable construction of plasma–facing components in a fusion reactor. Among thermal properties, thermal conductivity is one of the most important and is quite sensitive to radiation effects.[1]

In general, a large specimen is needed to measure the thermal conductivity, especially in so–called steady–state methods, where measuring procedures are complicated and time–consuming.[2] The development of the laser–flash method has improved the situations.[3] In development of highly thermally–conductive graphite in Japan[4], introduction of the standardized method of measuring their thermal conductivity with the laser–flash technique has been very effective to evaluate developed materials reliably.[4]

Nowadays, the laser–flash technique is a well–established method for measuring the thermal diffusivity, α, with very simple procedures in a wide temperature range. The thermal conductivity, κ, is evaluated by multiplying measured thermal diffusivity, α with the specific heat, c_p, and the density, ρ, as described in the following.

$$\kappa = \rho c_p \alpha \qquad (1).$$

The specific heat above room temperature is thought to be insensitive to small variation of compositions of material constituents and to radiation effects. The density could be measured easily. However, even in the laser–flash technique, the use of medium–size specimens(10mmϕ and 2–5mm thick) is necessary.

A heavy neutron irradiation is very expensive, thus the irradiation volume is usually limited. Furthermore, to reduce radioactivity of specimens, the specimens has to be as small as possible. Therefore, a miniature–specimen technique is essential for the study of heavy irradiation effects on fusion reactor materials. In this study, we developed a new laser–flash instrument which enabled us to measure the thermal diffusivity of small specimens. This paper will describe features of the developed instrument and show some preliminary experimental results.

2. Development of Instrument and Experimental Results

Primary target of the present research is to develop a laser flash instrument using a standard miniature specimen, so-called TEM(Transmission Electron Microscopy)-disk for heavy neutron irradiations. Size of the specimen is 3mmϕ and 0.15–0.5mm thick. In this study, we measured the thermal diffusivity of carbon-material and SUS 304, using specimens of different sizes by the developed instrument. The obtained results were also compared with those obtained by the conventional instrument. These measurements were carried out to clarify that the TEM-desk could be used for the measurement of thermal diffusivity by the present instrument.

One of the main restrictions concerning the geometrical size of the specimens is the finite time pulse effect[5]. A half-width of its laser-pulse is 0.3–0.4ms, and a pulse profile observed through its measuring electric circuits has a time-delay of about 1ms as shown in Fig. 1(a). Here, we measured a time interval between a center of the observed energy deposition profile and the time zero, when a trigger signal which activated the laser-pulse was detected. Also, the half-width of the measured laser pulse was 0.85ms, being about twice of that of the actual laser-pulse. So, phenomena only slower than a few tens milliseconds could be detected reliably in this system.

The response speed will be determined by response speeds of an infra-red temperature monitor and subsequent electric circuits for handling signals from the temperature monitor, and also by various time delays at electric interfaces in the system. In this study, we tried to improve the response speed of the electric circuits. The through rate of operational amplifiers in an amplifier circuit was increased and a time constant of the noise filter was minimized. Also, the isolation amplifiers in the amplifier were removed. Figure 1(b) shows the time-delay of the observed laser-pulse through the improved electric circuits. The time-delay was improved to be 0.53ms and the observed peak had a half-width of about 0.5ms, which was comparable with the half-width of actual laser pulse, about 0.4ms.

With increase of the response speed of electric circuits, the measured signals showed an exponential tailing. This tailing made a peak profile asymmetric and made a center of the energy deposition profile shift to a longer-time side. As one can see it clearly in Fig. 1(a), the peak observed in the conventional instrument revealed nearly symmetric, in contrast with that observed in the developed instrument. In the laser-flash technique, the initial response will be the most important and the measured tailing may not be important, although the tailing will affect details of obtained results. Then, we could examine the initial half part of observed peak profile. The observed peak position was 0.35ms and its half width is 0.45ms, which would be about the same energy-deposition profile of the actual laser-pulse.

Even though we improved the response-time of the electric circuits, we still had a limit of time resolution of about 0.5ms which came from the energy deposition profile of the conventional laser-pulse. Here, we tried to improve it by using a Q-switched laser. The Q-switched laser whose half width of the energy profile was 25ns was adopted as shown in Fig. 2. A energy-deposition profile measured in the present laser-flash instrument is also shown in Fig 1(c). The peak position was measured to be 0.05ms. So, in the present system, the $t_{1/2}$ value of about 2.5ms could be measured. From the following relation,

$$t_{1/2} = 0.139L^2/\alpha \quad (2),$$

we can estimate the minimum thickness of the specimen, L. Using thermal diffusivities at room temperature, the following thickness was estimated; 0.5mm, 0.1mm, 0.7mm, and 0.6mm for ATJ-graphite, SUS 304, copper, and aluminum, respectively. These values are well near the range of thickness of specimens irradiated in high flux reactors, up to high neutron doses.[6] Figure 3 shows measured temperature increase of highly conductive grassy carbon, GC-SS-J developed by Toyo Tanso Co Ltd., whose thickness was 0.535mm. The measured temperature increase had a half time, $t_{1/2}$, of 0.63ms, which was far out of the time–resolution of the conventional laser–flash instrument as described above.

Figure 4 shows measured values of thermal diffusivity of SUS 304 at room temperature as a function of specimen thickness. The diameter of the specimens was 3mm. The diffusivity of 0.0391cm^2/s is measured by a conventional laser–flash instrument for this specimen. Obtained values are well within + 10% of the recommended value in the range of thickness of 0.5–1.5mm. These values were obtained from the measured results, using the so–called $t_{1/2}$ method[3].

The $t_{1/2}$ method has many advantages but at the same time it has some setbacks caused by deviation of the measuring system from the theoretical descriptions.[7] The thicker specimens showed the larger thermal diffusivity as shown in Fig. 4. This implies that the heat flow parallel to a specimen surface and the inhomogeneous spatial distribution of energy of laser–flash will be its cause. The present laser flash was found to have inhomogeneous energy deposition profile as a result of attempt to focus the laser beam to a limited area. This attempt was done to increase spatial–density of the deposition laser energy, because the Q–switching initially caused decrease of the spatial energy density from about 6–10J/pulse to less than 1J/pulse. The specially designed specimen holder to shield the strong noise from the Q–switched laser would have also increased the heat flow parallel to the specimen surface.

More sophisticated analysis than the $t_{1/2}$ method will improve the situation, as the present system has a better time–resolution.[8] We preliminarily attempted to apply the so–called logarithmic slope method.[8&9] The obtained results are shown as closed diamonds in Fig. 4. Some improvement could be seen. However, further and detailed analyses must be needed to improve the present measurements.

Temperature dependence of the thermal diffusivity of 1 mm thick SUS 304 was measured on the specimens of 3 and 6mmϕ. The results are shown in Fig. 5, which also shows the results obtained on the 0.5mm thick specimen of 6mm. In general, the ob–

tained results fit the recommended value well within + 10% scatters. The trend that the present measurements gave higher thermal diffusivity at elevated temperature may be also due to the heat flow parallel to the specimen surface especially enhanced at elevated temperatures. Results that the 0.5mm thick specimens showed better fitting also support this speculation.

In general, the developed instrument could measure the thermal diffusivity of very thin specimens in a wide temperature range. However, we have one serious setback. The present laser has a power of about 1J per one pulse, smaller compared with the value of about 6J of the conventional instrument as shown in Table 1. However, the 25ns pulse means the energy power of $4x10^7$W, far larger than the energy density of $2x10^4$W of the conventional laser-flash instrument.

Optical radiation of such a high density laser pulse caused some surface modification, especially on the relatively low-melting point materials. Also, due to the high power pulse, the temperatures involved lead to a non linear behavior of heat transfer, resultantly, the data reduction method such as $t_{1/2}$ method would not be used. The change of surface conditions sometimes caused serious scatter of measured values. Some carbon-based materials showed increase of scatter with increase of number of measurements. Improvement of sensitivity of a temperature-monitor and optimization of time-width and strength of the laser-pulse will be needed.

III. Conclusion

A new Q-switched laser-flash instrument was developed for the measurement of thermal diffusivity of specimen having miniature-size suitable for neutron irradiation. The present instrument realized the time resolution better than 0.1 ms with a laser-pulse of 25 ns wide. Realization of measurements with a high time-resolution made it possible to measure the thermal diffusivity with thin specimens. It is expected that copper of 0.4mm thick, and SUS 304 of less than 0.1mm could be used for the measurements. In case of the ATJ graphite, 0.3mm thick specimen could be used for the reliable measurement. The preliminary measurements confirmed that the developed instrument could measure the thermal diffusivity reliably in the temperature range of 300–1300K, with the miniature specimens.

However, serious surface modification took place due to the deposition of high energy density, 1×10^9J/s, laser on measured specimen, which disturbed the measurements. Further optimization of the system will be needed.

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Table 1. Specifications of conventional and developed laser-flash instrument

	Conventional	Developed
Width of laser pulse	300 - 400 μs	25 ns
Laser energy per pulse	6 - 10 J	1 - 1.5 J
Diameter of homogeneous energy profile of laser pulse	10 mm	6 mm
Response time of measuring electrical circuits	about 1 ms	about 0.05 ms

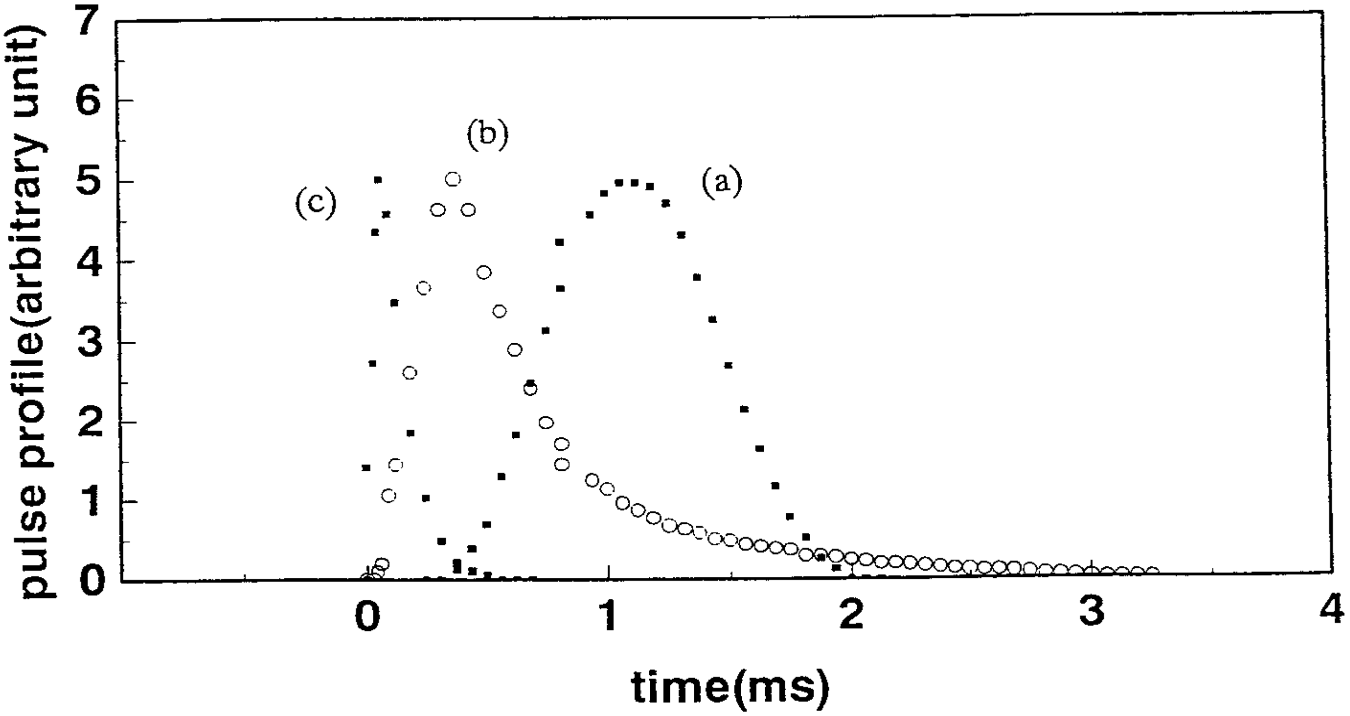

Fig. 1

Measured time-profile of laser-pulse.

(a) Conventional instrument,

(b) Developed instrument with normal laser-pulse,

(c) Q-switched laser-pulse.

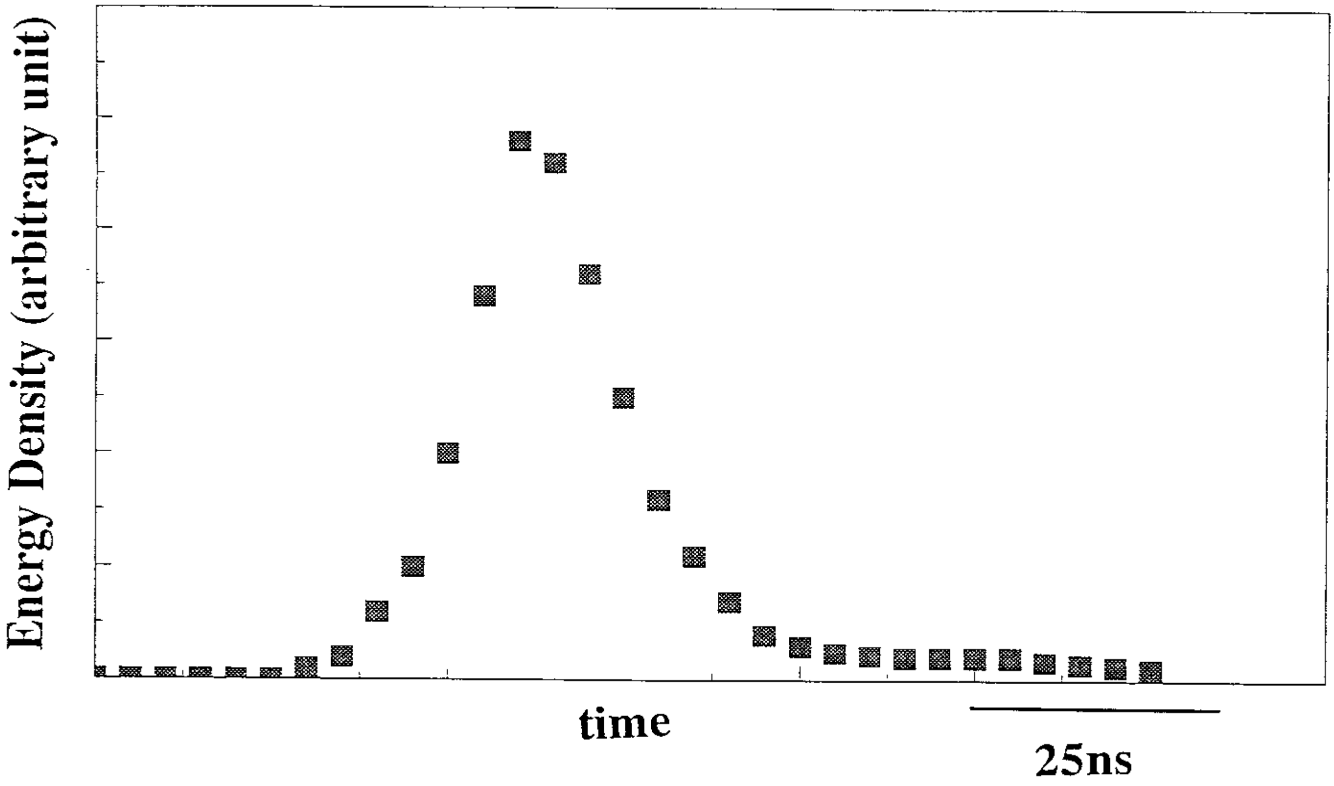

Fig. 2

Energy profile of Q-switched laser-pulse.

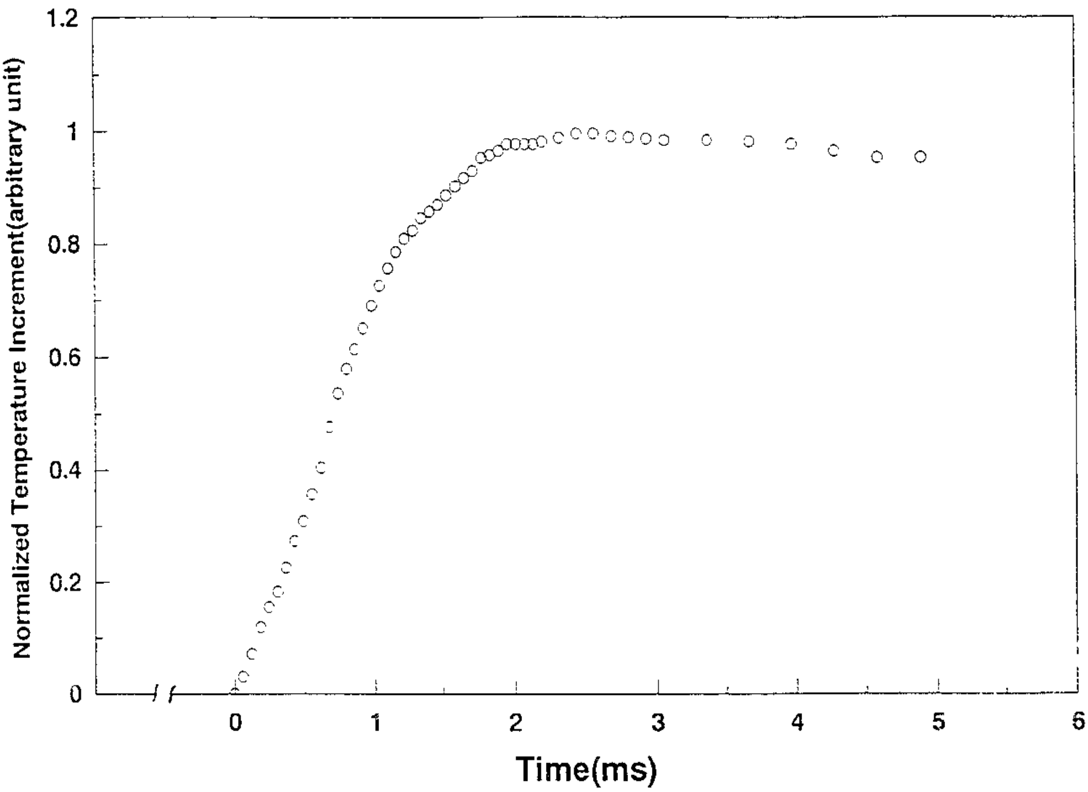

Fig. 3

Measured temperature change on GC-SS-J of 0.535 mm thick.

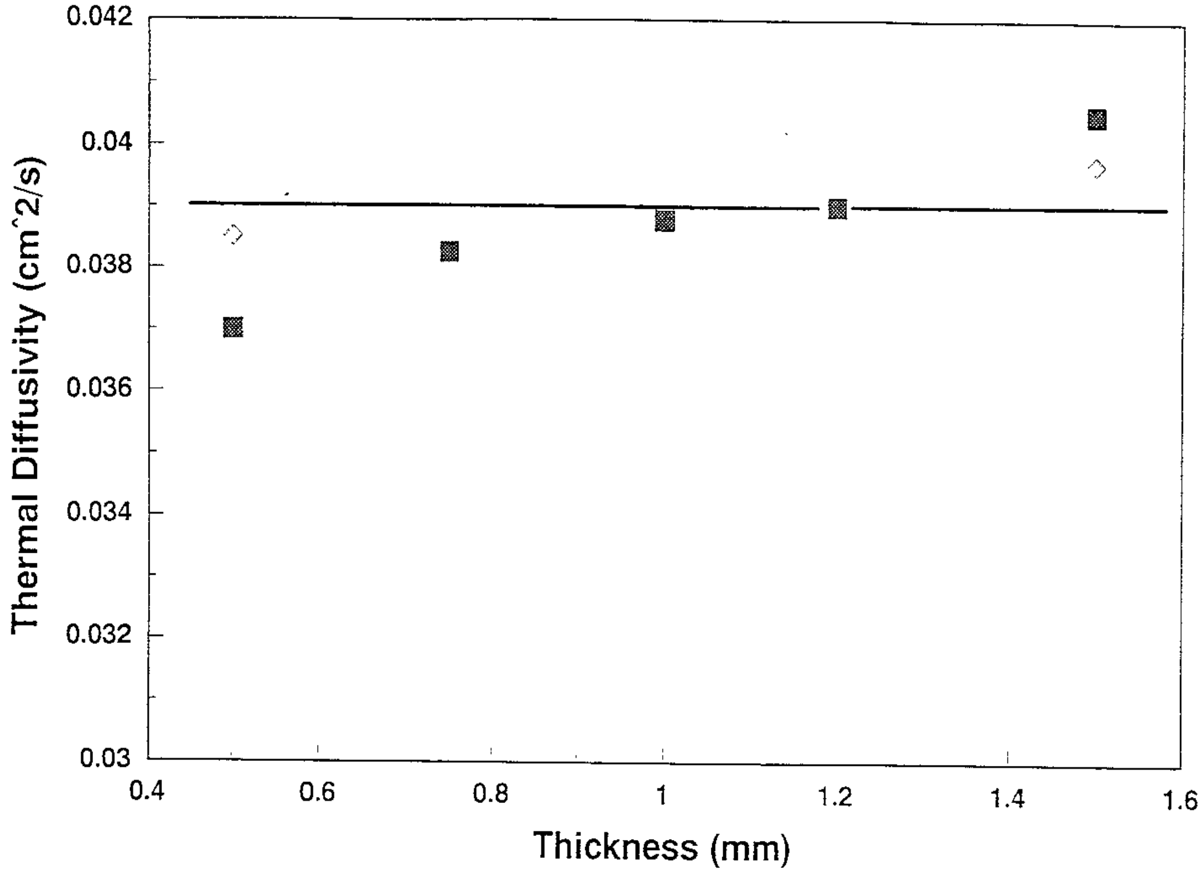

Fig. 4

Thickness dependence of measured thermal diffusivity on 3 mmϕ specimens;

closed square; obtained by the $t_{1/2}$method,

closed diamond; obtained by the logarithmic slope method.

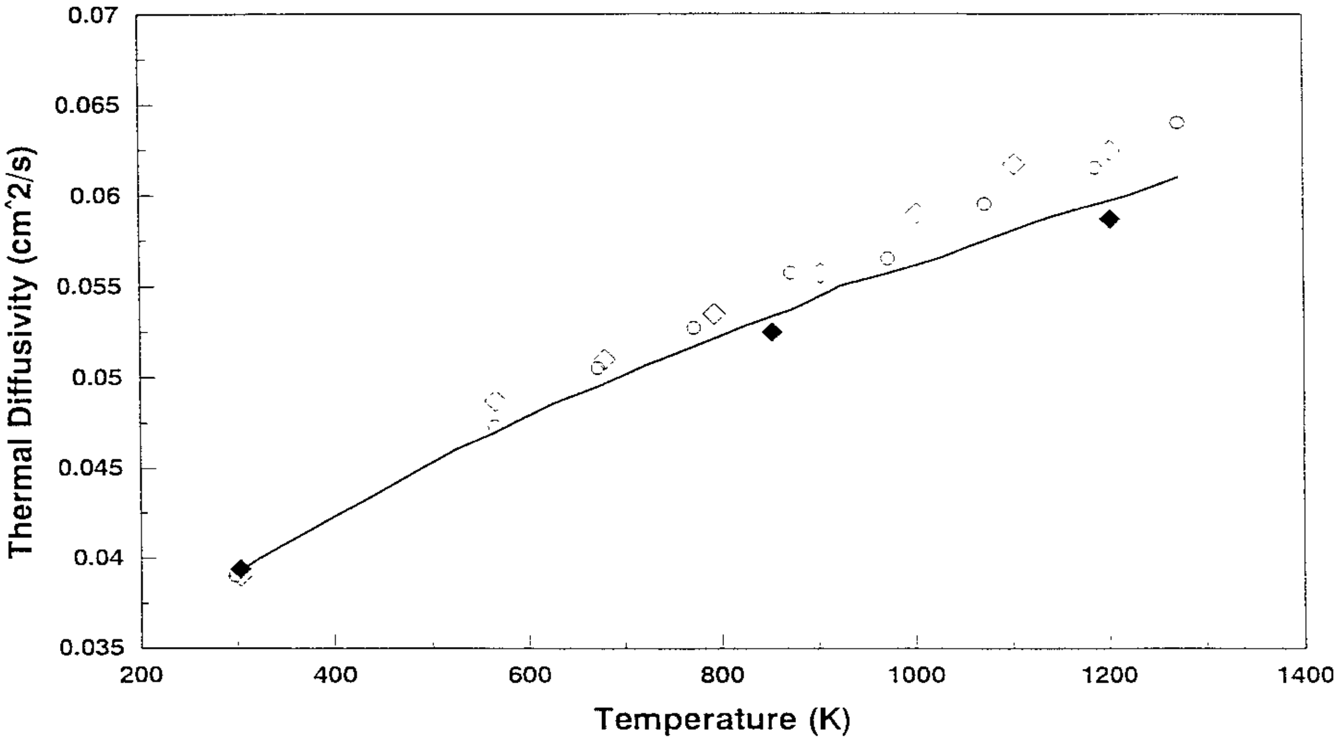

Fig. 5

Temperature dependence of thermal diffusivity measured on specimens;

open circle; 3 mmϕ and 1 mm thick,

open diamond; 6 mmϕ and 1 mm thick,

closed diamond; 6 mmϕ and 0.5 mm thick.

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