Systematic measurement of thermal diffusivity of $Pd_{40}Cu_{40-x}Ni_xP_{20}$ ($x=0,10,40$) alloys in liquid, glassy, crystallized, and supercooled liquid states by the laser flash method

Tsuyoshi Nishi,[1,*] Hiroyuki Shibata,[2] Hiromichi Ohta,[3] Nobuyuki Nishiyama,[4] Akihisa Inoue,[5] and Yoshio Waseda[2]
[1]*Graduate School, Department of Materials Processing, Tohoku University, 2-1-1, Katahira Aoba-ku, Sendai 980-8577, Japan*
[2]*Institute of Multidisciplinary Research for Advanced Materials, Tohoku University, 2-1-1, Katahira Aoba-ku, Sendai 980-8577, Japan*
[3]*Department of Materials Science, Ibaraki University, 4-12-1, Nakanarusawa, Hitachi 316-8511, Japan*
[4]*R&D Institute of Metals and Composites for Future Industries (RIMCOF), 2-1-1, Katahira Aoba-ku, Sendai 980-8577, Japan*
[5]*Institute for Materials Research, Tohoku University, 2-1-1, Katahira Aoba-ku, Sendai 980-8577, Japan*
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The availability of the thermal diffusivity values, α, of alloy liquids and their glassy states is very limited at the present time, although the thermal diffusivity values of alloy liquids might have a close relationship with their glass-forming ability. Thus, the purpose of this work is to determine the thermal diffusivity values of three particular alloys, $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$, in the liquid state, systematically obtained using a laser flash method involving a novel sample cell. In the liquid state, the thermal diffusivity value of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy showing an outstandingly high glass-forming ability is lower than that of the $Pd_{40}Ni_{40}P_{20}$ alloy and it is much lower than that of the $Pd_{40}Cu_{40}P_{20}$ alloy which has a low glass-forming ability. With respect to the thermal diffusivity values of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy, a positive temperature dependence was certainly obtained in all conditions: The glassy, supercooled liquid, and liquid states.

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INTRODUCTION

Pd-based glassy alloys with particular compositions, such as $Pd_{40}Cu_{40-x}Ni_xP_{20}$, are well known to exhibit glass transition with the appearance of a wide supercooled liquid region at over 80 K before crystallization.[1] The extremely high level of thermal stability of the supercooled liquid state enables the production of bulk amorphous alloys. The $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy especially has a high resistance against crystallization in a supercooled liquid state and exhibits an outstandingly high glass forming ability.[2–4] This glassy alloy has been seen to reach 75 mm in diameter.[2] On the other hand, the $Pd_{40}Ni_{40}P_{20}$ and $Pd_{40}Cu_{40}P_{20}$ glassy alloys are able to reach 25 and 5 mm in diameter, respectively,[5] within the best knowledge of the present authors.

Their high glass-forming ability and high thermal stability provide us a unique opportunity to investigate a number of physical properties of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ bulk amorphous alloy. For example, changes in viscosity[6] and electrical resistivity[7,8] have been found in its supercooled liquid state. Its specific volume,[9–11] crystallization behavior from the supercooled liquid state,[12] mixing heat,[13] local atomic structure,[14,15] and X-ray photoelectron spectroscopy analysis[16] have also been reported. However, the thermal diffusivity values of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ alloys are not available yet in any of the liquid, crystallized, and supercooled liquid states except for the glassy state[17] due to experimental difficulties.

On the other hand, the laser flash method[18] is widely employed and very versatile techniques for measuring the thermal diffusivity of various materials. The laser flash method has been found to overcome the experimental difficulties involved in measuring the thermal diffusivity of melts[19–21] at high temperatures by being coupled with the recently developed novel sample cell.

The main purpose of this work is to determine sufficiently reliable values of the thermal diffusivity of $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ alloys in the liquid state and to clarify the relationship of the thermal diffusivity in the liquid state with the glass-forming ability of Pd-based alloys metallic glass. Moreover, the thermal diffusivity values of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy were also obtained in the glassy, crystallized, and supercooled liquid states by means of the laser flash method.

EXPERIMENTAL

Master alloys were prepared by melting a mixture of nickel, copper, and pre-alloyed Pd-P ingots using an arc furnace under an Ar atmosphere. $Pd_{40}Cu_{40-x}Ni_xP_{20}$ master ingots of different compositions were then prepared by arc melting the mixtures of pure-Pd, -Ni, -Cu, and pre-alloyed Pd-P in an Ar atmosphere. In order to eliminate heterogeneous nuclei, a B_2O_3 flux treatment was carried out in a highly purified Ar atmosphere after alloy preparation. The molten $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ alloys were cast into copper molds to obtain these glassy alloys. The shape of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ glassy alloy was 50 mm in length and 5 mm in diameter, and the $Pd_{40}Ni_{40}P_{20}$ and $Pd_{40}Cu_{40}P_{20}$ glassy alloys in a plate shape was 12 mm in width, 24 mm in length, and 1 mm in thickness. The amorphous quality of the as-cast samples was checked by X-ray diffractometry employing Mo-Kα radiation. The as-cast samples of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ and $Pd_{40}Ni_{40}P_{20}$ were in a glassy state. The as-cast sample of $Pd_{40}Cu_{40}P_{20}$ was almost crystallized.

The $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ glassy alloys were heated using a tungsten mesh heater under a vacuum of less than 2×10^{-3} Pa. An appropriate sample cell must be used to maintain the $Pd_{40}Cu_{30}Ni_{10}P_{20}$,

$Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ in a liquid state and $Pd_{40}Cu_{30}Ni_{10}P_{20}$ in a supercooled liquid state. This sample cell consisted of an alumina tube sandwiched by two sapphire plates.[19–21] Sapphire is known to be transparent to Nd:glass lasers and infrared rays. This sample cell is able to effectively measure the thermal diffusivity of molten iron, cobalt, nickel[19] and germanium.[21] In regard to volume expansion on melting, the cell is fixed by a graphite fixture at room temperature.[19–21]

The thermal diffusivity values of $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ alloys in a liquid state were measured using a vertical-type laser flash apparatus. The molten sample was placed in the sample cell.[19–21] The upper surface of a sample having a disk shape was instantaneously irradiated through the upper sapphire plate by a Nd:glass laser (10 J, 1060 nm emission wavelength). The temperature increase at the sample's bottom surface was measured through the lower sapphire plate by using an InSb infrared detector (1.2–5.5 μm effective wavelength). The temperature gradient induced by laser flash heating in the liquid sample is negative to a gravity force. Therefore, the density-driven convection in the liquid sample does not take place under this experimental condition. Based upon the theory proposed by Carslaw and Jaeger,[22] the thermal diffusivity, α, can be evaluated using the following equation:[18]

$$\alpha = 0.1388\frac{l^2}{t_{1/2}}, \quad (1)$$

where l is the sample thickness and $t_{1/2}$ is the time required for the back surface of the sample to reach one half of the maximum increase in temperature.

When the sample cell is used to measure the thermal diffusivity of the alloys in the liquid state, it is necessary to consider the effect of not only the radiative heat loss but also the conductive heat loss at the interface between the sample and cell material in order to obtain a reliable value of thermal diffusivity. When these two heat loss factors become significant, the temperature of the back surface of the sample reaches its maximum and decreases along the line proportional to the form of $\exp(-kt)$ as schematically shown in Fig. 1(a), where k is the coefficient of the temperature decrease and t is the elapsed time after a laser irradiation. The value of k was obtained from the analysis of the measured temperature response curves in the attenuation curve region. In the present work, the maximum value of k was found to be 0.01 at most. Accordingly, it was concluded that the effect of two factors of heat loss are negligibly small under the present experimental condition.[20] Thus, the thermal diffusivity values in the liquid state, in the glass state, and in the supercooled liquid state were estimated using Eq. (1).

The sample cell was not used for the measurement of the thermal diffusivity of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ in the crystallized state. The temperature response curve of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy in the crystallized state is shown in Fig. 1(b). It was again concluded that the influence of the radiative heat loss was negligibly small under the present experimental condition because the obtained value of k in the crystallized state was much smaller than that of the liquid state. Thus, the thermal diffusivity value of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy in the crystallized state was also estimated using Eq. (1).

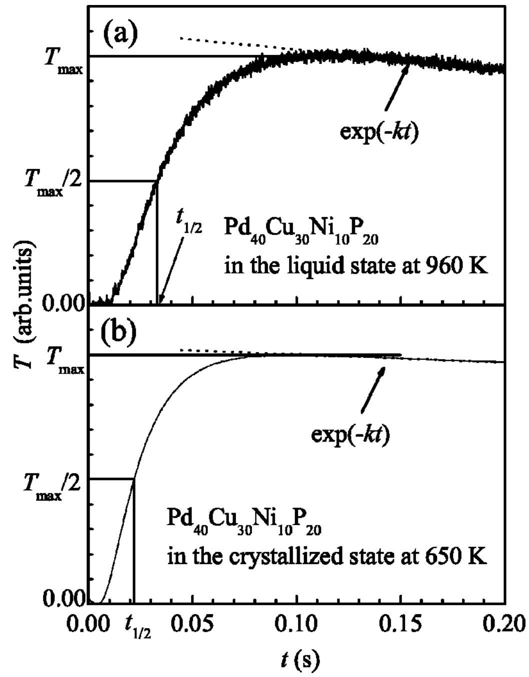

FIG. 1. Temperature response curves of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ in a liquid state at 960 K and in a crystallized state at 650 K.

In the supercooled liquid and liquid state, the sample was heated to 1120 K at a heating rate of 0.17 K/s and cooled to 720 K at a cooling rate of 0.17 K/s. Before laser irradiation, the temperature of the sample was maintained at the desired temperature for about 15 min in order to stabilize the temperature during both the heating and the cooling periods.

In the glassy state, the thermal diffusivity of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy was obtained in the temperature range from 300 to 585 K during heating of about 0.17 K/s because the crystallization of the glassy sample must be repressed during the course of the measurement.

In order to obtain a crystallized sample, the liquid sample was cooled to room temperature at a cooling rate of 0.2 K/s because the critical cooling rate for the crystallization of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy without the flux treatment was 1.58 K/s.[23] Measurement of the crystallized sample was performed from 300 to 760 K. The experimental procedure was identical to that used in the case of the glassy state.

RESULTS AND DISCUSSION

The thermal diffusivity values of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy were measured in the temperature range from 920 to 1120 K. For the $Pd_{40}Ni_{40}P_{20}$ alloy, the measurements in the liquid state were made in the temperature range from 1000 to 1120 K, and for the $Pd_{40}Cu_{40}P_{20}$ alloy in the temperature range from 1060 to 1120 K. As shown in Fig. 2, the high degree of reproducibility of the experimental thermal diffusivity data was confirmed by repeating the measurements under a given condition three times.

The thermal diffusivity values (unit: m^2/s) of the three alloys, $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$, in the

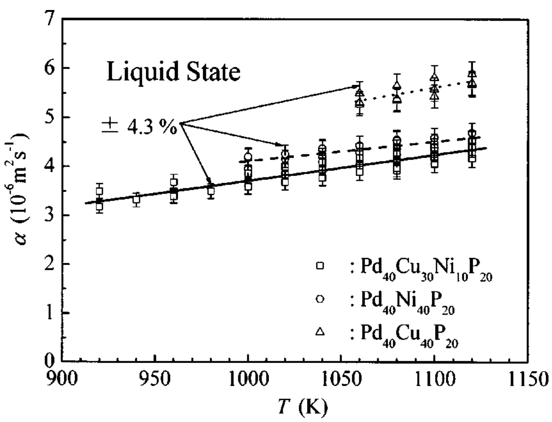

FIG. 2. Thermal diffusivity of $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ in a liquid state as a function of temperature. The symbols $\square$, $\bigcirc$, and $\triangle$ denote the experimental data while their linear fitting results are shown by lines.

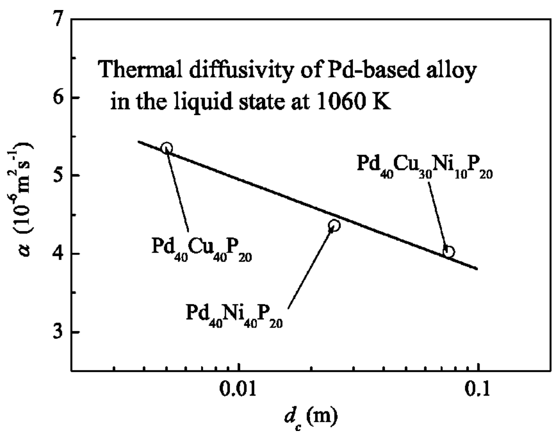

FIG. 3. Thermal diffusivity of $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ in the liquid state at 1060 K as a function of the critical diameter, d_C, of glass formation.

liquid state are summarized in the following equations as a function of temperature, T (unit: K).

$$\alpha_{Pd_{40}Cu_{30}Ni_{10}P_{20}} = 5.32 \times 10^{-9}(T - 920) + 3.28 \times 10^{-6} \quad 920 \leqslant T \leqslant 1120 \tag{2}$$

$$\alpha_{Pd_{40}Ni_{40}P_{20}} = 3.95 \times 10^{-9}(T - 1000) + 4.12 \times 10^{-6} \quad 1000 \leqslant T \leqslant 1120 \tag{3}$$

$$\alpha_{Pd_{40}Cu_{40}P_{20}} = 6.67 \times 10^{-9}(T - 1060) + 5.35 \times 10^{-6} \quad 1060 \leqslant T \leqslant 1120 \tag{4}$$

The experimental uncertainty is estimated to be at most ±4.3%. This includes the experimental errors due to the signal to noise ratio in the temperature response curve and the thermal expansion of the cell materials used for estimating the sample thickness.[19,21] The thermal diffusivity values of the liquid $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$ alloys show a positive temperature dependence, although the origin of this behavior cannot be identified with certainty at the present time. In the liquid state, the thermal diffusivity value of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy which has an outstandingly high glass-forming ability is lower than that of the $Pd_{40}Ni_{40}P_{20}$ alloy and it is also found to be much lower than that of the $Pd_{40}Cu_{40}P_{20}$ alloy which has a low glass-forming ability in the liquid state, as shown in Fig. 2.

A positive temperature dependence of the thermal diffusivity data has been generally found for molten pure metals[19–21] as well as for liquid metallic glass alloys. Thus, the temperature dependence of the thermal diffusivity data may not be directly related to the glass-forming ability of bulk metallic glass. However, the low thermal diffusivity values are considered to relate to glass-forming ability. The order of the thermal diffusivity values among the three alloys investigated is discussed in view of the knowledge of their atomic structures in the liquid state. The atomic ordering structure is quite likely to exist in Pd-based liquid alloys, and such an ordering structure is also suggested in their glass state.[15] For example, the trigonal prism structure consisting of six metallic atoms (Pd or Ni) around P has been suggested in the $Pd_{40}Ni_{40}P_{20}$ glassy alloy.[24] On the other hand, X-ray diffraction results for the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ metallic alloy indicate a polyhedral ordering structure consisting of eight or nine metallic atoms (Pd or Cu or Ni) coordinated around P.[14] It is not too much to say that Pd, Cu, and Ni atoms are bonded not only with P but also with metallic elements, because the interactions between Pd and Cu have large negative mixing heats.[25] Some free electrons in the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ liquid alloy are contributed to the P atoms bonding with Pd, Cu, and Ni atoms, and Pd with Cu coordinated to P. Thus, the number of conduction electrons, which are not contributed to the atomic ordering structure in the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy, is considered to be less than those in the $Pd_{40}Ni_{40}P_{20}$ and $Pd_{40}Cu_{40}P_{20}$ alloys. The conduction electrons dominate the heat transport in Pd-based alloys because the Wiedemann-Franz law has been recognized.[26] Therefore, the number of the free electrons may be related to the order of the thermal diffusivity values. As a result, the thermal diffusivity values of the liquid alloys examined here diminish in the order of $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$, as shown in Fig. 2.

The following point is noteworthy. The critical diameter, d_C, of glass formation is 75 mm for $Pd_{40}Cu_{30}Ni_{10}P_{20}$, 25 mm for $Pd_{40}Ni_{40}P_{20}$, and 5 mm for $Pd_{40}Cu_{40}P_{20}$.[2,5] The lower the thermal diffusivity values of Pd-based alloys in the liquid state at 1060 K, the larger the critical diameter of glass formation becomes, as easily seen in Fig. 3.

In order to measure the alloys' thermal diffusivity in the supercooled liquid state, the liquid sample was cooled to below liquidus, continuously. The thermal diffusivity values of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ in the glassy, crystallized, supercooled liquid and in the liquid state were also measured in the temperature range from 300 to 1120 K. The results are shown in Fig. 4 together with those in the liquid state. Characteristic temperatures such as the glass transition temperature, T_g, crystallization temperature, T_x, eutectic temperature, T_e, and liquidus temperature, T_l, of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ glassy alloy[1] are given in Fig. 4. Since the measurements of

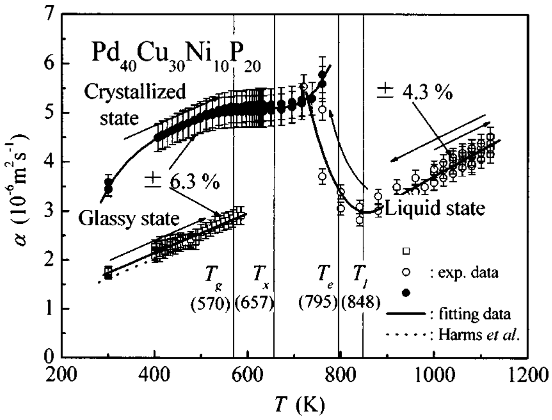

FIG. 4. Thermal diffusivity of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ in the glassy, crystallized, supercooled liquid, and liquid states as a function of temperature.

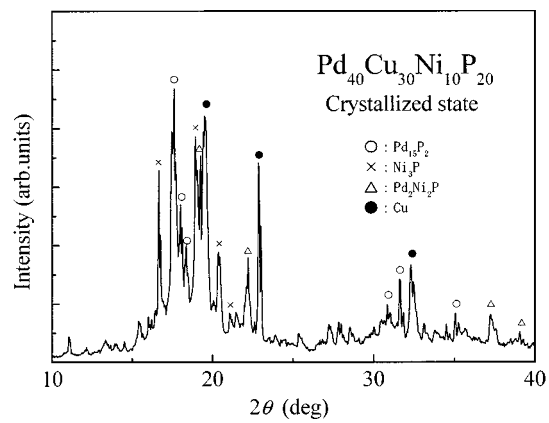

FIG. 5. X-ray diffraction patterns of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ in the crystallized state.

$Pd_{40}Cu_{30}Ni_{10}P_{20}$ in the glass and crystallized states were performed during heating, the degree of uncertainty of thermal diffusivity in the glassy and crystallized states is larger than that in the supercooled liquid and liquid states. Nevertheless, the experimental uncertainty of the present thermal diffusivity values in the glass and crystallized states in the temperature range from 300 to 670 K is approximately ±6.3% at most.

The thermal diffusivity values of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy show a positive temperature dependence in the glassy state. It is noted that the thermal diffusivity values of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy in the glassy state were measured by Harms *et al.*[17] using the thermal-wave technique; they show close agreement with the present results as seen in Fig. 4, although their data are limited to the lower temperature range.

The values measured at a temperature just below T_l (840 K) correspond to those measured in the supercooled liquid state. These values are approximately equal to the value (2.9×10^{-6} $m^2 s^{-1}$) at T_g. The structure and essential mechanism of thermal diffusion in the supercooled liquid state is presumably unchanged in the temperature range from T_g to T_l. This is consistent with the structural features obtained by X-ray scattering.[14,15] The thermal diffusivity values were measured downward from 840 to 720 K at a cooling rate of 0.17 K. In this temperature region, the thermal diffusivity values rapidly increase with decreasing temperature, and this variation corresponds to the volume fraction of the crystalline and liquid phases.

The thermal diffusivity value of the crystallized state is much larger than that of the glassy case, as shown in Fig. 4. On the other hand, Fig. 5 shows the X-ray diffraction patterns (Mo-Kα radiation) of the crystallized $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy. The crystalline phases of crystallized $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy were identified as the mixture of $Pd_{15}P_2$, Ni_3P, Pd_2Ni_2P, and Cu. Data regarding the thermal properties of $Pd_{15}P_2$, Ni_3P, and Pd_2Ni_2P phases is not available. However, such distinct differences in the thermal diffusivity values of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloys may be attributed to the structural difference between the crystallized state with atomic periodicity and the glassy state with nonperiodicity.

CONCLUSION

The thermal diffusivity values of three particular alloys, $Pd_{40}Cu_{30}Ni_{10}P_{20}$, $Pd_{40}Ni_{40}P_{20}$, and $Pd_{40}Cu_{40}P_{20}$, in the liquid state were systematically determined using the laser flash method involving the described sample cell. The clear differences in their thermal diffusivity values suggest that thermal diffusivity may be one of the indicators of the glass-forming ability of Pd-based glassy alloys.

The thermal diffusivity values of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy in the glassy, crystallized, and supercooled liquid states have also been obtained. The results are summarized as follows:

(1) The thermal diffusivity value at 840 K in the supercooled liquid state is approximately equal to the value at the glass transition temperature;

(2) A positive temperature dependence is confirmed in the thermal diffusivity values of $Pd_{40}Cu_{30}Ni_{10}P_{20}$ in the glassy and liquid states;

(3) The thermal diffusivity of the $Pd_{40}Cu_{30}Ni_{10}P_{20}$ alloy in the crystallized state is much larger than that in the glassy state.

The lower the thermal diffusivity values of Pd-based alloys in the liquid state, the larger the critical diameter of glass formation clearly becomes. Thus, the present authors maintain the view that the thermal diffusivity values of alloy liquids might be an indicator of their the glass forming ability.

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*Present address: Department of Materials Science, Japan Atomic Energy Research Institute, 2-4, Shirakatashirane, Tokai-mura, naka-gun 319-1195, Japan.

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