放射熱交換と放射測温技術を用いた熱物性計測技術に関する研究

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A Study on Measurement Techniques for Thermophysical Properties Using Radiative Heat Exchange and Radiation Thermometry

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ABSTRACT:

Thermophysical properties (the thermal conductivity, specific heat capacity, emissivity, etc.) are a group of properties which are closely related to transfer and accumulation of heat. Recently, numerical simulation techniques have been rapidly developed in designing various devices where thermal problems are critical. In response to the development in simulation techniques, thermophysical properties, including those under special condition (high temperatures, small/thin samples, etc.), have become more important as basic parameters for the simulation.

On the other hand, the most of the traditional methods are referred as "adiabatic method", in which a sample is insulated carefully with insulators and guard heaters. Hence, measurement apparatuses in these methods were usually large and complicated, and the time needed for a measurement consequently became longer. In addition, ideal thermal insulation was difficult at higher temperatures due to enhanced radiation heat loss which increases in proportion to the absolute temperature to the fourth power.

In this study, a pair of techniques, i.e., radiative heat exchange and radiation thermometry, have been newly employed to develop measurement methods for thermophysical properties. Instead of using insulators, the amount of radiative heat exchange between the sample surface and the environment in vacuum is evaluated precisely based on the Stefan-Boltzmann law. The temperature of the sample surface is measured remotely with a radiation thermometer. Based on these techniques, two different measurement methods for thermal conductivity, and a method for specific heat capacity and emissivity at high temperatures using a pulse-heating technique have been developed. These methods enable measurements of thermophysical properties with a smaller sample, with a more simple apparatus, in a shorter measuring time, and at higher temperature ranges compared with those in the traditional methods. A thermograph is also used in the measurement of the thermal conductivity to obtain the temperature distribution on the sample surface. The use of the thermograph enables to obtain the thermal conductivity even from a non-linear temperature distribution on a small and thin sample that is inappropriate in the traditional methods. The details of the each measurement method are described below.

Firstly, a method for the measurement of the thermal conductivity using radiative heat exchange has been developed in which a steady heat flux exists in the direction of length of a small ribbon-shaped sample (approximately, $0.5\times5\times40$ mm^3). The sample is held by a pair of heated holders in vacuum, and is also Joule-heated by passing a direct current through itself (Figs. 2-1 and 2-2). A temperature distribution along the sample caused by thermal conduction and radiative heat exchange is measured by a thermograph, and the thermal conductivity of the sample is computed using eq. (2-7). This technique is most applicable for high-conductive materials such as metals (more than about 10 W$\cdot$m^{-1}K^{-1}). Using this method, the thermal conductivity of stainless-steel (SUS 304) was measured in the temperature range of 50-600 °C (Fig. 2-6). An error analysis including FEM (finite element method) calculations shows the inaccuracy in the thermal conductivity is less than ± 2 % (at a standard deviation) in the lower temperature range of 50-300 °C.

Secondly, another method for thermal conductivity has been developed in which a heat flux exists in the direction of thickness of a plate sample (approximately, $2\times25\times25$ mm^3). The sample is embedded on the

surface of a metal block with high thermal conductivity and coated with black paint (Fig. 2-12). Additional heater on the sample surface may be used for more accurate evaluation of the hemispherical total emissivity of the black paint. The sample assembly is kept at a constant higher temperature in vacuum (Fig. 2-13). A temperature difference between the surfaces of the sample and the metal block caused by the difference in thermal conductivity is measured by a thermograph, and the thermal conductivity of the sample is computed using eq. (2-13). This technique is most applicable for low conductive materials such as polymers and ceramics (0.1~10 $W \cdot m^{-1}K^{-1}$). The thermal conductivities of silicone rubber, Pyrex glass, and Pyroceram were measured in the temperature range of 30-110 °C (Figs. 2-20, 2-21, and 2-27). An analysis for inaccuracy including FEM calculations shows that the inaccuracy in thermal conductivity is less than ± 2 %.

Thirdly, a method for the specific heat capacity and hemispherical total emissivity of electrically-conductive materials using a pulse-heating technique has been developed. In this method, a ribbon sample is heated rapidly (in 100~300 ms) from room temperature to a temperature close to the melting point by passing a large direct current (1000~3000 A). During the experiment, the heating current, a voltage drop between voltage probes contacted on the sample surface, and the sample temperature (with a high-speed radiation thermometer) are measured. The specific heat capacity and the hemispherical total emissivity of the sample are computed based on the heat-balance equation (3-1). In order to decide the true surface temperature, the spectral emissivity of the sample surface is determined with a hemispherical mirror. Using this method, the specific heat capacity and the hemispherical total emissivity of graphite materials including C/C (carbon-carbon) composites were measured (Figs. 3-10 and 3-11).

After the primary development, an advanced pulse-heating technique with a feedback-control of the heating current has been developed. This technique is based on rapid heating of a sample up to a preset high temperature and subsequent keeping of that temperature for a brief time period (about 500 ms). In order to maintain a constant temperature, the heating current was adjusted rapidly (every 100 μs) by a computer-controlled feedback-control system based on the sensing signal supplied by a high-speed radiation thermometer. A high-speed laser-ellipsometer is also employed for the determination of the normal spectral emissivity of the sample surface. In the temperature plateau maintained by the feedback-control technique, the hemispherical total emissivity of the sample is obtained from the heat-balance equation (3-10) assuming that the heat loss due to thermal conduction to the sample holders is negligible in such a short-time experiment. This technique enables to obtain the hemispherical total emissivity in smaller data scatter because the measured quantities are averaged in the plateau over a longer time period compared to those in the traditional pulse-heating method. Using the present technique, the specific heat capacities and the hemispherical total emissivities of the three refractory metals, tantalum, molybdenum, and tungsten, were measured (Figs. 3-21, 22, 23, 27, 28, and 29). An analysis for inaccuracy shows that the inaccuracies in specific heat capacity and hemispherical total emissivity are less than ± 2 % and ± 3 %, respectively at 2200 K. For more details of the measurement methods developed in this study, refer to the other publications shown in chapter 8.1 (page 97).