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Measurement of fluid thermal conductivity using a micro-beam MEMS sensor

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ABSTRACT

A new method for measuring thermal conductivities of gases and liquids was established by demonstrating the measurement of five kinds of liquid and air. It uses a sensor named “micro-beam sensor” that is a ~10-μm-long free-standing platinum membrane suspended across a trench on a silicon substrate and heated in a sample by DC. This method is unique in that it is a steady-state measurement but free from the effect of natural convection owing to the micrometer size of the sensor. Improving the method for precisely determining the temperature of the sensor and modifying the device from those used in our previous feasibility study, we successfully measured the thermal conductivity ranging from ~0.03 to ~0.6 W/(m·K) within 4% error.

1. Introduction

A variety of methods have been proposed for measuring thermal transport properties of gases and liquids. Among them, the transient hot-wire method, which uses a thin wire as a heat source and a temperature detector in a fluid sample, is one of the most widely used methods [1–4]. With this method, the thermal conductivity and the thermal diffusivity are determined from an analytical solution to one-dimensional radial heat conduction equation around a wire. To minimize the error associated with the effect of heat loss from both ends of the wire, or to take its effect into account, the double-wire method [5] and the short-hot-wire method [6] have been respectively developed and successfully used for measuring various kinds of fluids [7–10].

Although these methods are useful, at least 100–200 mL of sample must be prepared for the transient hot-wire method and ~40 mL for the short-hot-wire method. We therefore proposed a new method using a MEMS sensor to significantly reduce the volume of sample [11,12], which is favorable particularly for measuring valuable fluids such as rare chemical compounds and biological samples. The sensor is composed of a ~10-μm long free-standing platinum membrane suspended across a trench on a silicon substrate, and was named “micro-beam sensor.” We heat this sensor by a constant DC in a sample and determine the consequent temperature rise from the measured electrical resistance. A unique feature of this method is that the measurement is done at a steady state. Owing to its size, *i.e.* only ~10 μm in length, the temperature of the sensor reaches a steady state within 1 ms [11]. In addition, the effect of natural convection is negligible because the Rayleigh number is extremely small. Thus the measurement is simple, finished within a short time, but requires no high-time-resolution instrument.

A variety of MEMS sensors have been proposed for measuring thermal transport properties of gases and liquids. Kuntner et al. and Ernst et al. [13–15] developed a micromachined device consisted of a heater and membrane thermistors for measuring thermal conductivity and thermal diffusivity of fluids. Cruz et al. [16] designed a microfabricated thermal conductivity detector for chemical sensing. Atherton et al. [17] designed a thin-film-thermocouple based sensor to measure the thermal conductivity of a microliter-volume liquid. Zhang et al. [18] designed a microcalorimeter for thermal characterization of liquids and polymer thin films. Udina et al. [19] and Cheng et al. [20] fabricated micromachined thermoelectric sensors for testing natural gas and liquids. All these sensors have complex structures that consist of various patterns of micro-heaters and temperature detectors fabricated on a freestanding membrane. The thermophysical properties are then determined from a numerical analysis of heat conduction for a physical model that precisely represents the structure and thermophysical properties of the sensor. In contrast, the principle of our measurement is much simpler and more robust because the uniformly heated free-standing membrane works as a heat source and a temperature detector. In addition, the physical properties of the sensor per se, which affect the accuracy of measurement, are precisely determined during the measuring process.

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Following a theoretical study that has proposed the methodology [11], we demonstrated that the temperature of the prototype sensor did depend on the thermal conductivity of surrounding samples; the temperature rise was ~70% higher in the air than in FC-72 [12]. The paper was important in a sense that it demonstrated the feasibility of the method for the first time. However, the result was insufficient because the measured temperature rise was significantly different from what predicted by the theoretical analysis. We therefore extensively re-examined the whole measuring process to find the cause and came up with the idea that the effect of voltage drop at the electrode pad must be taken into account in determining the temperature of the sensor. Another weak point of the prototype sensor from the practical point of view was the fact that a silver paste was used to connect lead wires with electrode pads. This was the reason why only a perfluorocarbon liquid and air were used as samples in our feasibility study. In the present study, we solved all these problems and measured thermal conductivity ranging from ~0.03 to ~0.6 W/(m·K) using five kinds of liquid and air. The significantly improved result demonstrated the completion of the method for measuring thermal conductivities of liquid and gases.

2. Experiments

2.1. Micro-beam sensor

The micro-beam sensor was fabricated on a 10 mm × 10 mm silicon substrate through the protocol described in our previous paper [12]. The sensor was a 9.3-μm-long, 0.49-μm-wide and 40-nm-thick free-standing platinum membrane suspended across a trench as a part of integrated pattern with four electrode pads (Fig. 1). Because of undesired etching of the substrate underneath the electrode pads during fabrication of a trench, ~3 μm wide membrane overhung the trench at the both ends of the sensor. The sensor chip was annealed at 220 °C for 3 h in a vacuum before use to stabilize the electrical and thermal properties of the platinum film.

2.2. Experimental apparatus

We improved the measuring device from the prototype [12] to make it more robust and useful for wide application. The silicon chip with a sensor on its top surface was mounted on an aluminum plate at the bottom of a ceramic holder (Fig. 2). A thermistor was placed on the aluminum plate near the chip to precisely measure the temperature of the chip. Four electrode rods with a sharp tip connected with a lead wire at another end were pressed onto the terminal pads using springs to make secure electrical contact at the resistance smaller than a few milliohm. This connection with no silver paste enables the device to be used in water and the other solvents. The holder was set in a ~100-mL Teflon sample container, and immersed in a thermostat bath. Although the sample necessary for the measurement is less than a micro-liter in principle, the sensor was dipped in a container in the present study because the main objective was to demonstrate the measurement.

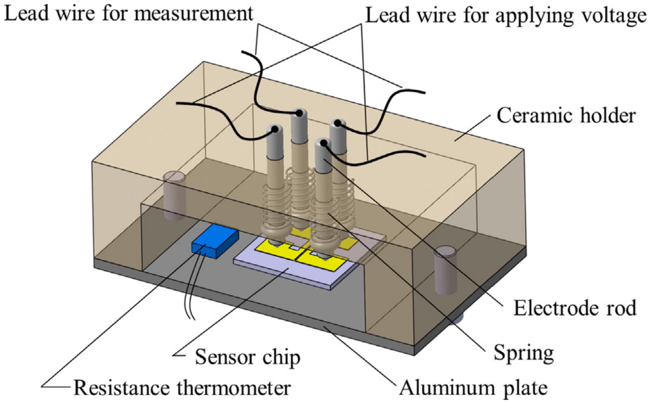

Fig. 2. Measuring device.

The electrical resistance of the sensor was measured by a four-terminal method. A constant current was supplied from a DC power supply (Keithley 2601A) to heat the sensor through two external wires connected to the terminal pads. The voltage drop at the sensor was measured between two internal terminal pads. The voltage drop at a 100-Ω standard resistor connected in the power line was also measured to determine the electric current. The measurement was carried out at five different heating rates by stepwise increasing the current, collecting ten data at 660-ms intervals using two digital multi-meters (Keithley 2002) at each current. The experiment was replicated three times. To protect the sensor from burnout due to an accidental current, two short-cut circuits with switches were opened only during the measurement.

Prior to the experiment, the sensor was calibrated by placing the sensor in a vacuum chamber. The electrical resistance of the sensor was measured at several current intensities keeping the chip at a given temperature.

3. Numerical analysis

3.1. Heat conduction analysis

The thermal conductivity of a sample fluid is determined from comparison of measured temperature rise of the sensor with the theoretical solution of heat conduction from the sensor to the surrounding fluid. To this end, three-dimensional analysis was con-

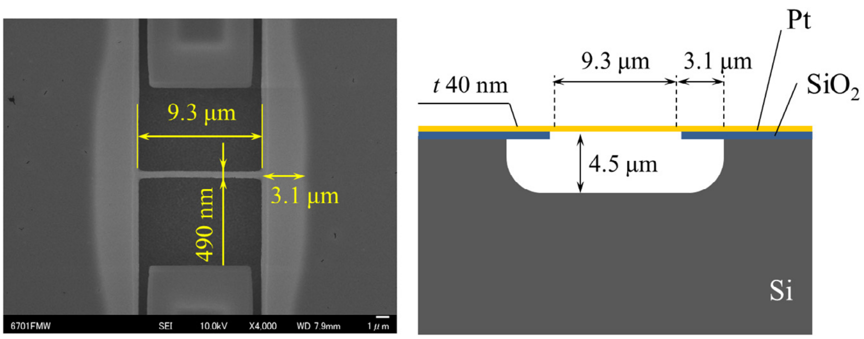

Fig. 1. The SEM image and the cross section of the sensor.