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Micro-beam sensor for detection of thermal conductivity of gases and liquids

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ABSTRACT

A prototype of "micro-beam" MEMS sensor that is made of a thin metallic film suspended across a trench on a silicon substrate was fabricated for examination of the feasibility of detecting thermal conductivity of gases and liquids. Heating the sensor in a sample demonstrated the potential measurement at a steady state because no natural convection took place during heating. The measured temperature rise of the sensor agreed fairly well with the temperature rise estimated by a numerical analysis of heat conduction to a sample fluid from the sensor with given measured dimensions. The temperature of the sensor was significantly higher in the air than FC-72 as well, indicating the feasibility of detecting thermal conductivity by the proposed method.

1. Introduction

The transient hot-wire method is one of the most popular techniques for measuring thermal transport properties of gases and liquids [1–4]. The method is based on an analytical solution to the one-dimensional radial heat conduction equation around a wire that is heated in a fluid sample. Hence a long, thin wire, typically 100–200 mm long and 10–50 μm in diameter, has been used as a heater/sensor to reduce the effect of heat loss from both ends of the wire. In addition, the end effect that results in axially non-uniform temperature in the wire was compensated by utilizing two wires with different lengths and analyzing the difference in the temperature rise of these wires [5]. In contrast, Fujii et al. [6] used significantly shorter wire, approximately 10 mm long, and solved a two-dimensional heat conduction equation to evaluate axial temperature distribution. A unique feature of their method, named a 'short-hot-wire' method, is that the temperature at both ends of the wire does not change during the measurement because of a large heat capacity of terminal electrodes compared with a heating power. The method has been applied successfully for measuring molten salts [7], polymers [8] and low density gases [9,10]. The advantage of the short-hot-wire method over the conventional hot-wire method is that the volume of sample could be reduced from 100–200 ml to ∼40 ml.

While most of the studies on thermophysical properties have been devoted to reduce the uncertainty of the measurement, there is a demand for *in situ* measurement of thermophysical properties of fluids [11]. Since MEMS sensors have a potential to be used for the *in situ* measurement of small samples with accuracy adequate for the purpose, various types has been proposed. Ernst et al. [12] and Kuntner et al. [13] fabricated a heater and germanium thermistors on a silicon-nitride membrane and measured thermal conductivity and thermal diffusivity of gases and liquids. Zhan and Tadigadapa [14] and Udina et al. [15] used a thermopile and polysilicon heater on a freestanding membrane for measurement of thermal conductivity. Cheng et al. [16] designed a CMOS chip to measure thermal diffusivity of liquids. Iervolino et al. [17] fabricated a calorimeter chip with a complex two-membrane structure for measuring thermal conductivity and thermal diffusivity of liquids. A micro-hot plate [18] and a micro-thermal conductivity detector [19] were also proposed for a gas sensor or a chemical sensor. A common feature of these sensors was that a heater and temperature detectors were fabricated separately on a freestanding membrane, while they have their specific structure and configuration. Thermal transport properties of fluids are determined by comparing measured temperature response with the solution to a transient three-dimensional heat conduction equation for each specific system.

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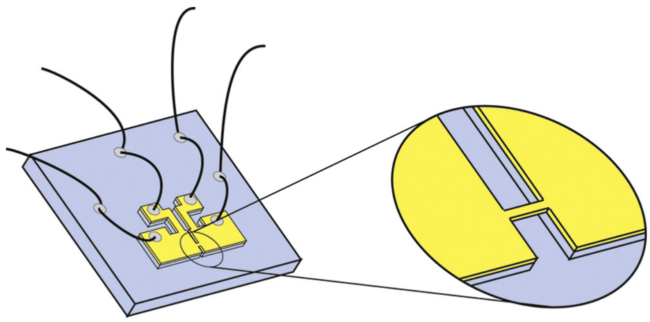

Fig. 1. Schematic of micro-beam sensor.

In contrast to these separate-type sensors, the authors have proposed a "micro-beam MEMS sensor" made of a thin metallic film that is suspended from a silicon substrate across a trench on a silicon substrate. The basic idea of the method was the same as the short-hot-wire method where the sensor was suspended from electrodes that have large heat capacities. Significant differences are that the sensor is three orders of magnitude smaller than the short hot wire and has a rectangular cross section. As a feasibility study, we have conducted a three-dimensional heat conduction analysis around the sensor [20]. The numerical simulation showed that the temperature in the sensor, typically 10 μm long, increased rapidly after applying the power to the sensor, and reached a steady state within 1 ms, which is presumably much faster than the occurrence of natural convection. The average temperature of the sensor was higher in lower-thermal-conductivity fluids as well. Based on these results, we have proposed a new steady-state method, and demonstrated with numerical simulations that the thermal conductivity was determined within an error of several percent.

The objective of the present study is to demonstrate the feasibility of the method using prototype micro-beam sensors. It is probable that the configuration of the fabricated sensors is different from that assumed in the simulation due to fabrication processes. It is therefore important to demonstrate that the temperature rise of the sensor depends on the thermal conductivity of sample fluids. It is also important to evaluate the difference in the temperature rise of the sensor between the experiment and the numerical simulation.

The proposed method is, as far as we know, the only method that measures the thermal conductivity of gases and liquids at a steady state within an extremely short time. It does not require a high-speed measuring instrument that is used in the transient methods. We are able to determine the thermal conductivity, in principle, by a single measurement of the temperature rise of the sensor. In addition, the beam-type sensor that is used for a heater and a temperature detector has much simpler structure than many other miniaturized sensors. The principle of the measurement is therefore simpler and more robust. Most of the proposed methods are the comparative measurement based on the calibration using some standard samples. In the calibration, the difference between the measurement and the theoretical analysis is to be taken into account by some models and coefficients. The robust and simpler principle of the measurement is favorable for introducing appropriate coefficients from phenomenological consideration.

2. Experiments

2.1. Micro-beam sensor

A sensor and terminals were fabricated on a 10 mm × 10 mm silicon substrate as shown in Fig. 1. In the present study, two sensors, platinum and gold sensors, were fabricated with similar processes. The pattern shown in Fig. 1 was drawn first by an electron beam lithography on an EB resist that was spun-coated on a Si(1 0 0) substrate with a SiO_2 layer on the surface. A platinum film or a gold film was then deposited by the EBPVD method after deposition of titanium (8 nm thick) as an adhesion layer. The suspended beam-type sensor was fabricated by a lift-off technique. The EB resist and SiO_2 layer were removed by wet-etching processes, i.e. immersion of the chip in a liquid resist-remover and subsequently in a buffered hydrofluoric acid. Then the silicon substrate was partly etched using CF_4 gas to fabricate a trench to suspend the platinum/gold film from the substrate. The adhesion layer was also removed during the wet-etching process. Finally, the chip was exposed to oxygen plasma for cleaning the surface of the sensor and forming an oxidized insulation layer on the silicon surface. Before use, the sensors were annealed in a vacuum chamber at 220 °C for 3 h.

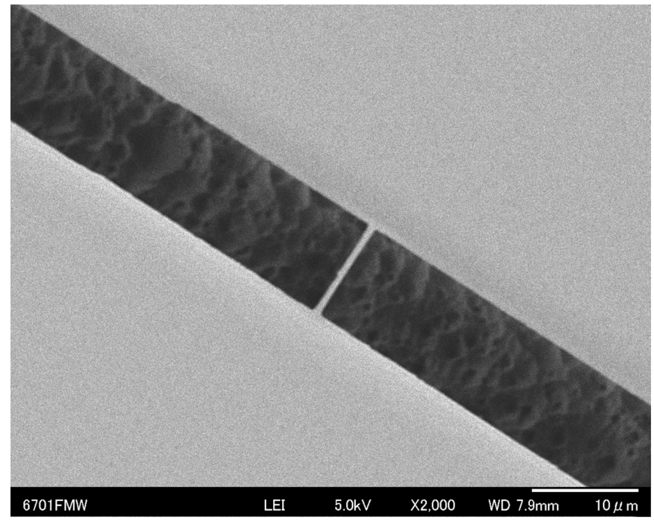

Fig. 2. SEM image of a platinum sensor.

Fig. 2 shows a SEM (scanning electron microscope) image of the platinum sensor. The configuration of sensors was determined by using a SEM, an AFM (atomic force microscope) and a LCM (laser confocal microscope). Because of the etching process for fabrication of a trench, the substrate underneath the deposited electrode was partly eroded and thus the sensor was protruded from an overhang of a platinum/gold film at both sides of the trench. The overhang part was distinguished from the film on the substrate in a high resolution SEM image by the difference in the brightness (Fig. 3). The sizes of the sensors (length L_s, width W_s, thickness D_s, width of overhang L_r and depth of trench H_t) were summarized in Table 1. Although a deeper trench and smaller overhang are preferable for the measurement, both requirements are not compatible because a longer time of etching results in a deeper trench and a wider overhang. As a preliminary study, we have emphasized the trench depth in the fabrication of the platinum sensor and a smaller overhang in the gold sensor. This is the reason why two sensors showed significant differences in the overhang width and the trench depth, while the lengths were similar (∼10 μm).

Table 1
Dimensions of fabricated sensors.

	Platinum sensor	Gold sensor
Length L_s (μm)	9.80	9.18
Width W_s (μm)	0.64	0.39
Thickness D_s (nm)	43	68
Overhang width L_r (μm)	6.60	3.15
Trench depth H_t (μm)	7.3	0.8