Bridge-Based Microsensor for Determining the Thermal Properties of Liquids

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Due to unique features like small thermal masses and reduced conductivities, miniaturized thermal sensors offer properties superior to those provided by comparable macroscopic measurement setups. In this contribution a micromachined device is applied to characterize the thermal transport properties of various liquids. By means of sinusoidal excitation of a heater structure placed on a micro-bridge, and recording the resulting temperature at a specified distance using integrated germanium thermistors also located on micro-bridges, the liquid's thermal parameters can be determined. A simple two-dimensional analytical model allows to interpret the amplitude and the phase of the measured sinusoidal temperature variation, yielding both, the thermal conductivity and the diffusivity of the liquid.

Introduction

In the past decades, a number of different measurement methods for thermal material properties of liquids have been developed. Most devices comprise of a single heater or a heater in combination with a spatially separated temperature sensor [1] – [3]. In the former case the temporal evolution of the heater temperature is recorded, whereas in the latter case the temperature in some distance from the heater is measured using the additional temperature sensor. In both approaches the resulting temperature response is determined by the thermal material properties of the surrounding medium and can thus be utilized to extract these parameters. Regardless of the particular realization, all methods aim to restrict an imposed thermal flow to the liquid under test, and to avoid spurious thermal shunts. Due to the unique geometrical features that can be achieved by microtechnology, micromachined devices offer various opportunities for fulfilling this requirement. Here, thin membranes are most commonly applied to decouple the actual sensing region from the sensor substrate and thus obtain high sensitivity and a short response time. For some liquids, however, the remaining spurious heat flow in the membrane is still not negligible. These unwanted thermal shunts can be further decreased by utilizing structures, like cantilevers or micro-bridges. If, on the other hand, materials are used which prohibit exceeding a particular maximum temperature in subsequent process steps, the manufacturing of such sophisticated devices can be challenging. A prominent example is the application of amorphous materials, where high temperatures would cause recrystallization. In this contribution a bridge-based microsensor with highly sensitive amorphous germanium thermistors for determining the thermal conductivity and diffusivity of liquids is presented.

Sensor Design and Fabrication

The sensor presented in this contribution consists of three silicon nitride bridges supported by a silicon frame. On the outer bridges highly sensitive amorphous germanium thermistors (T2 and T3) are located, whereas on the central bridge a chromium heater (H) is placed. Amorphous germanium exhibits high values of both, resistivity and associated temperature coefficient. The specific resistivity is about 5 Ωm and the temperature coefficient of resistance is approximately –1.8%/K at room temperature. Additional thermistors (”substrate thermistors” T1 and T4) arranged at the silicon frame supporting the bridges provide the opportunity of determining the ambient temperature (see Fig. 1 and Fig. 2).

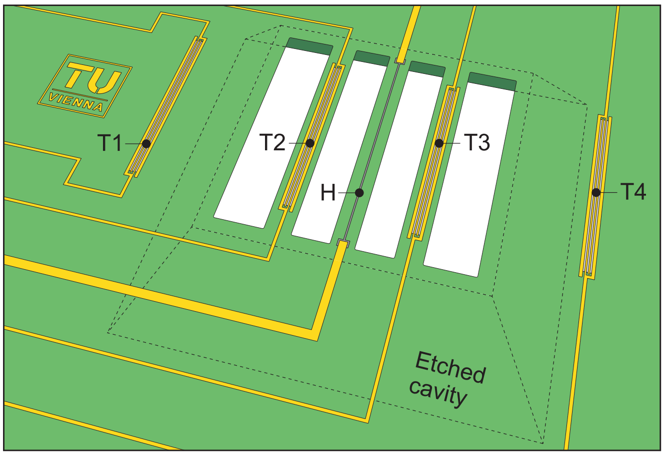

Fig. 1: Schematic picture of the device.

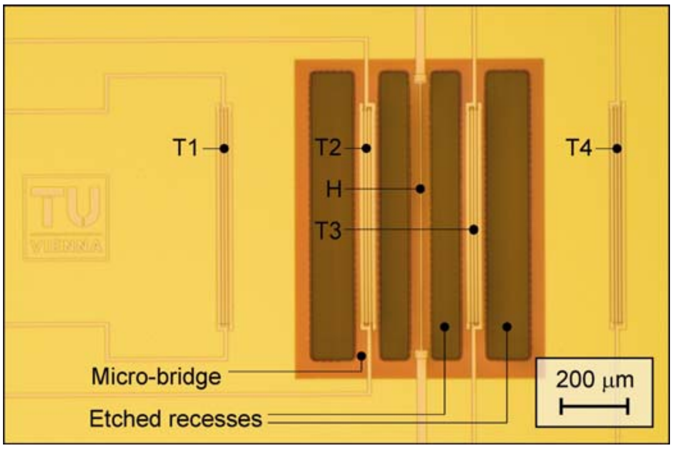

Fig. 2: Photomicrograph of the device.

To fabricate the bridge-based sensor, a 350 µm thick, (100) oriented, and double-sided polished silicon wafer was used. The wafer was coated with 250 nm of thermally grown silicon oxide (SiO_2) and 70 nm of LPCVD silicon nitride (Si_3N_4) on both sides. First, a 250 nm thick layer of germanium has been vapor-deposited and patterned using lift-off technique to form the thermistors. Next, a 130 nm thick layer of chromium has been applied and patterned to create the heater. Subsequently, a titanium-gold-chromium layer featuring a thickness of 50-100-30 nm has been deposited and lift-off patterned to obtain interdigitated electrodes for the thermistors, and connection leads to the bonding pads for heater and thermistors. Then, a low stress silicon nitride (SiN_x) protective film with a thickness of about 1000 nm has been applied using low temperature plasma enhanced chemical vapor deposition (PECVD) at 100 °C. Here, the low deposition temperature prevents the germanium film from recrystallization. Afterwards, square apertures in the wafer backside coating were created by means of photolithography and reactive ion etching (RIE). The membranes have then been manufactured using a KOH based anisotropic wet etching process. In order to obtain the required micro-bridges, the membranes have subsequently been patterned from the topside using photolithography and a RIE process. The apertures for the bond pads were made in the same step. Finally, the chromium has been removed from the bond pad areas by means of a wet-etching. Consequently, the sensor chips feature an overall thickness of about 1.3 µm.

Measurement Principle and Setup

By applying a sinusoidal heater current, a diffusive heat wave propagates from the heater into the surrounding liquid. The steady-state amplitude ΔT_{pk}[1] and the phase Φ of the temperature oscillations measured by the thermistors are determined by the thermal properties of the liquid. The phase Φ denotes the phase lag between the applied heating power and the resulting temperature oscillation. Moreover these temperature oscillations are related to the applied heating power and thus feature twice the frequency of the applied AC heater current plus an additional DC component. Considering the geometry of the heater structure, the corresponding AC temperature field can be approximated by using the solution of the two-dimensional heat diffusion equation for a periodic line source $P(r,t)=P_0 e^{j\omega t}$ yielding

$$\Delta T(r) = \frac{P_0}{2\cdot\pi\cdot\lambda}\cdot K_0\left(\sqrt{\frac{j\omega}{a}}\cdot r\right). \tag{1}$$

Here P_0 denotes the peak of the AC component of the heating power per unit length of the heater structure, λ the thermal conductivity, K_0 the modified Bessel function of the second kind, ω the angular frequency, a the thermal diffusivity, and r the radial distance from the line source [4]. The thermal diffusivity a is related to the heat capacity c_p by

$$a = \frac{\lambda}{\rho\cdot c_p}, \tag{2}$$

where ρ denotes the mass density. It can be seen, that the amplitude in Equation (1) is essentially determined by the thermal conductivity of the liquid under investigation whereas the corresponding phase is governed by the thermal diffusivity. Note that the model above considers heat conductance only, which means that the effects of heat radiation and convection, i.e. flows induced by the non-uniform temperature distribution, are neglected. This can be justified since the considered excess temperatures are in the range of fractions of degree Centigrade. For the measurements, the device was completely immersed into the sample liquid and a sinusoidal heater voltage was applied. In order to single out the steady-state amplitude ΔT_{pk} and the phase Φ of the temperature oscillation measured at the thermistors, a DC voltage was applied to the thermistors and the resulting current oscillations were measured. After eliminating the superposed DC-component, representing the ambient temperature plus the temperature increase associated with the DC-offset of the generated heating power, the amplitude and the phase of the exciting signal's second harmonic were determined by means of a lock-in amplifier (see Fig. 3).

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[1] Here, Δ indicates the excess temperature, i.e., the difference between ambient and actual temperature.

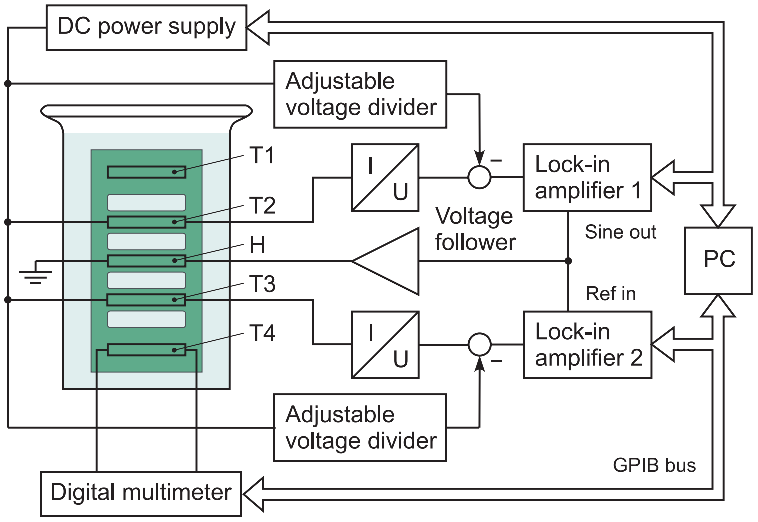

Fig. 3: Measurement setup.

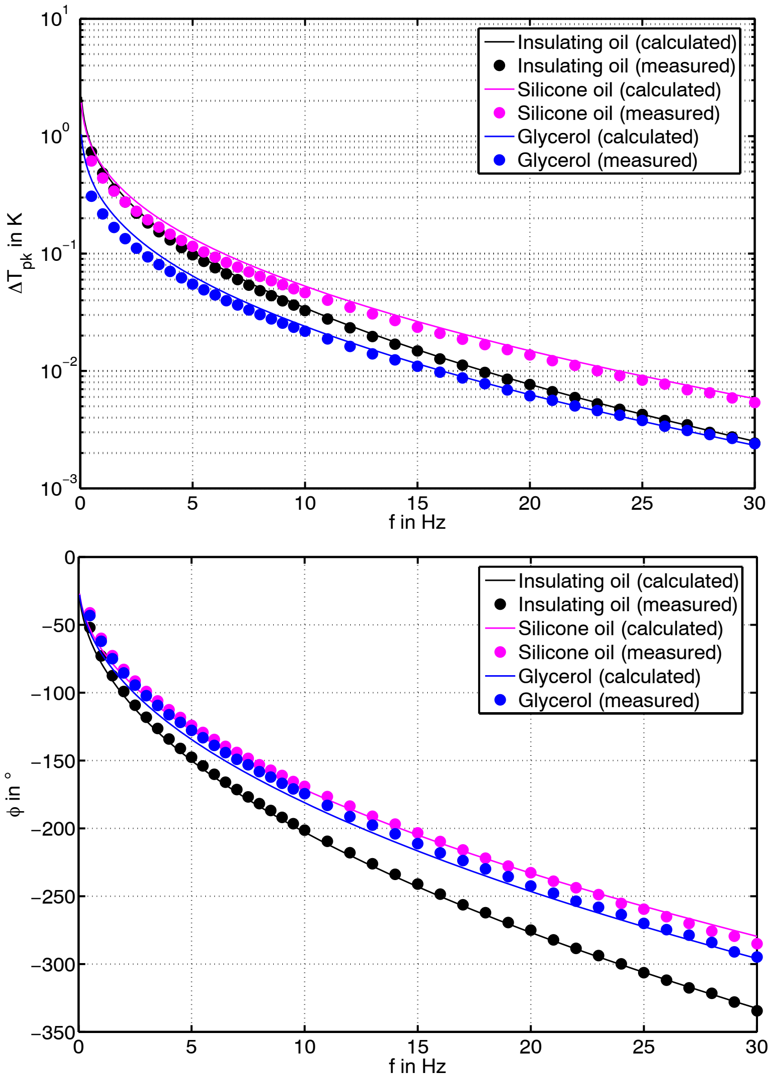

Fig. 4: Amplitude ΔT_{pk} and phase Φ measured for a heater voltage of 1 V_{rms}.

Results and Discussion

In order to investigate the performance of the developed sensor, different liquids have been analyzed. The liquids have been chosen in such a way, that a comparatively large parameter range in both, the thermal conductivity and the thermal diffusivity could be probed. Figure 4 shows the amplitude ΔT_{pk} and the phase Φ of the AC excess temperature amplitude measured at the thermistor T3 compared to the values for the model described above versus the frequency *f*.

It can be seen that the measurement results correspond well with those predicted by the simple two-dimensional model. Consequently the developed device enables to determine the thermal conductivity and diffusivity of liquids without the need for a complex model for data interpretation.

Conclusion

By utilizing a micromachined structure, the simultaneous measurement of the thermal conductivity and diffusivity of an adjacent liquid has been demonstrated. This was achieved by applying a sinusoidal heater signal and recording amplitude and phase of the resulting temperature increase in some distance by means of a germanium thermistor located on a micro-bridge. A simple two-dimensional analytical model was used to interpret the measurands. The presented approach is well suited for thermal liquid analysis in the laboratory and in the field. In particular, it is suited for online monitoring applications, where size and power consumption can be issues and where the determination of the absolute values is secondary compared to the detection of relative changes.

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References

[1] J. J. Healy, J. J. de Groot, and J. Kestin: “The theory of the transient hot-wire method for measuring thermal conductivity”, Physica C, Vol. 82, no. 2, 1976, pp. 392-408.

[2] S. E. Gustafsson, E. Karawacki, and M. N. Khan: “Transient hot-strip method for simultaneously measuring thermal conductivity and thermal diffusivity of solids and fluids”, Journal of Physics D: Applied Physics, vol. 12, no. 9, 1979, pp. 1411-1421.

[3] D. G. Cahill: “Thermal conductivity measurements from 30 to 750 K: the 3ω method”, Review of Scientific Instruments, vol. 61, no. 2, 1990, pp. 802-808.

[4] H. S. Carslaw and J. C. Jaeger: “Conduction of Heat in Solids”, Clarendon Press, 2nd ed., 1986.