Gas micro-flow-metering with the in-channel Pt resistors

Jan M. Łysko, Bogdan Latecki, and Marek Nikodem

Abstract— Standard thermo-conductive gas flow meters have the side-channel integrated with the temperature detector and heater coils, both winded around the tube. This design suffers from a high thermal capacity, reduced sensitivity in the lower limit flow range, high thermal inertia, long response time and necessity to amplify electronically the output signal. The newly designed TCD detector can be applied as a precise gas flow meter. To identify the composition of the unknown gas, the TCD unit requires a connection to the separation column, application of the reference channel and highly stable flow rate regulator. To measure flow rates, the same TCD unit requires only one flow channel application, with active resistors inside it, and a definition of the gas type. In this work principles of the TCD design, technology and flow rate sensitivity tests are presented.

Keywords— detector, silicon, gas, flow, thermo-conductivity.

1. Introduction

The thermal conductivity detector (TCD) unit was designed and fabricated for applications related to systems of multicomponent gas mixture detection [1–4]. TCD performance requires initial separation of the gas mixture components, stable carrier gas flow rate and two parallel flow channels of highly symmetrical geometry. This device can also be used as a precise gas flow meter. In this mode the gas type has to be determined. The most advantageous feature of the presented device is the location of the resistors inside flow channels and highly reduced geometrical dimensions of metal paths. Resistors were patterned in a thin layer Pt/Cr sandwich by means of an advanced microelectronics technology. Direct contact between the resistors and the investigated gas, as well as extremely low thermal capacity of the resistors, improve sensitivity, output signal level and response time.

2. Design

The TCD unit was designed (Fig. 1) and fabricated with the application of standard microelectronics CAD software, tools and technology, supplemented by several technological steps that are typical of silicon MEMS [2–4]. The resistors were patterned using the Pt/Cr lift-off technique on a silicon substrate covered by PECVD SiO_2/Si_3N_4 layers. Grooves were etched in a (100) silicon substrate in a TMAH + water solution, with the SiO_2/Si_3N_4 layers serving as the etching mask. The TCD unit consists of a glass plate with parallel groves milled with a diamond-disk blade. During the alignment and bonding of the silicon chip and the glass plate, horizontal flow channels were formed at the silicon-glass interface. Flow-channel openings at the opposite edges of TCD were connected to the external gas tubing. The application of microelectronics technology resulted in low dimensions of the device elements, symmetry, repeatability of physical and geometrical parameters, as well as the satisfactory yield of correctly manufactured device chips.

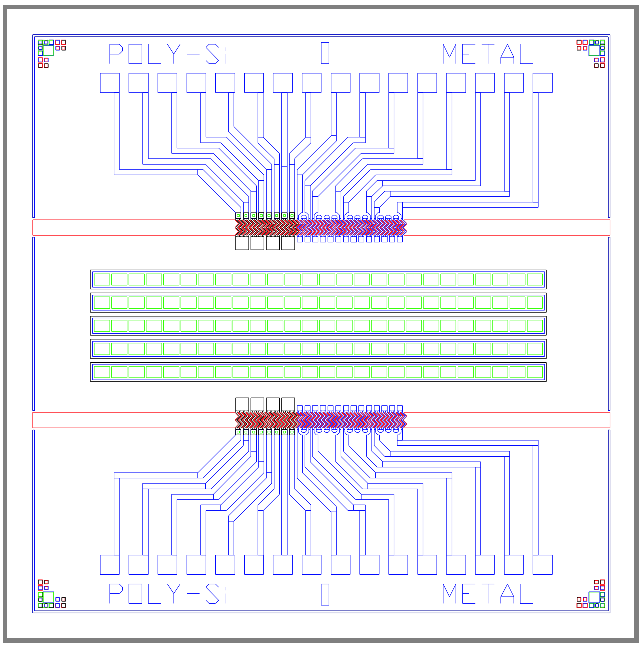

Fig. 1. TCD mask layout.

3. Technology

The most critical steps of the process of TCD silicon chip fabrication are lithography of Pt resistors and groove etching. Resistors were formed on a 3-inch, (100) Si wafer covered with SiO_2/Si_3N_4 PECVD layers. Lift-off technique and negative ma-N-1420 resist were used to pattern metal (4000 Å Cr/Pt layer) paths. A chromium pad of 50 Å is used to improve the adhesion of metal paths to silicon nitride. Grooves in the silicon substrate were defined by the next photolithography mask. The mask contained multiple shapes oriented in the directions $[0\ \bar{1}\ 1]$ and $[0\ 1\ \bar{1}]$ and located between the resistor paths. Mask shapes were etched first in the silicon nitride and silicon dioxide layers (Fig. 2a). After resist removal, wafers were diced into

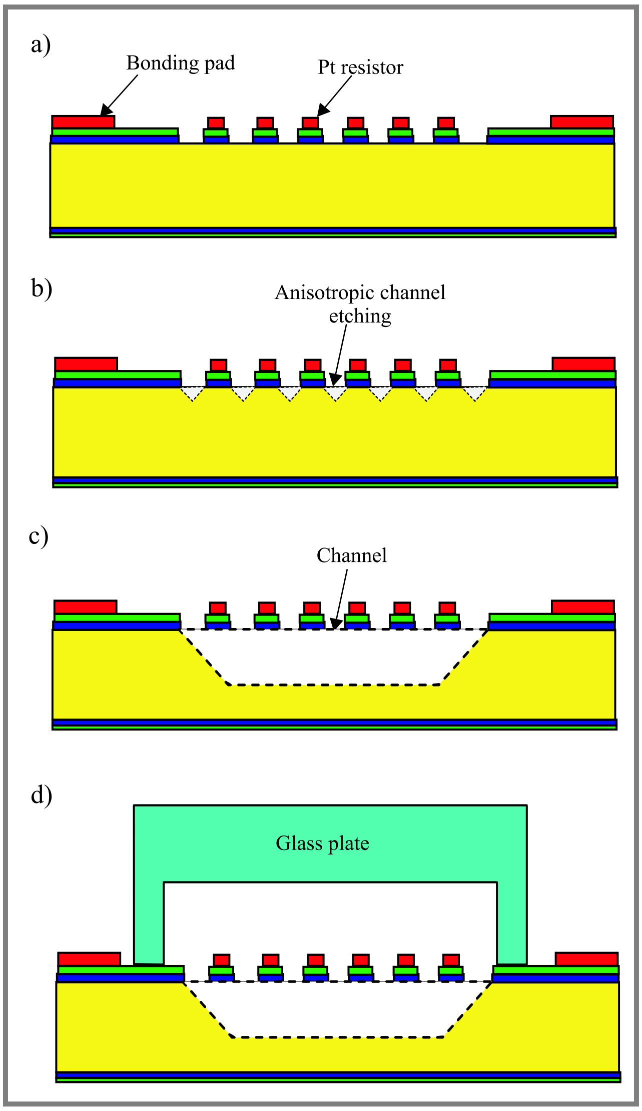

Fig. 2. Schematic drawings of the TCD chip cross section: (a) after the dielectric layer and Pt layer deposition, photolitography of the Pt resistor and photolitography of the channel; (b) initialization of the silicon substrate anisotropic etching; (c) final silicon chip with the resistor paths suspended accross the channel; (d) after silicon-to-glass anodic bonding step.

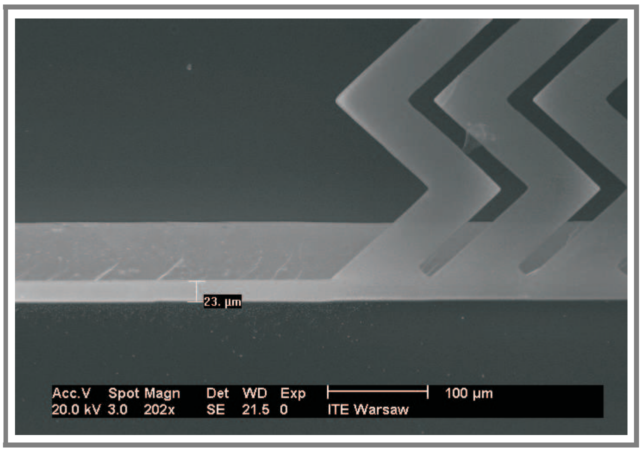

Fig. 3. SEM of a TCD silicon chip in the groove edge region. Lateral etch range in the direction [111] is 23 μm.

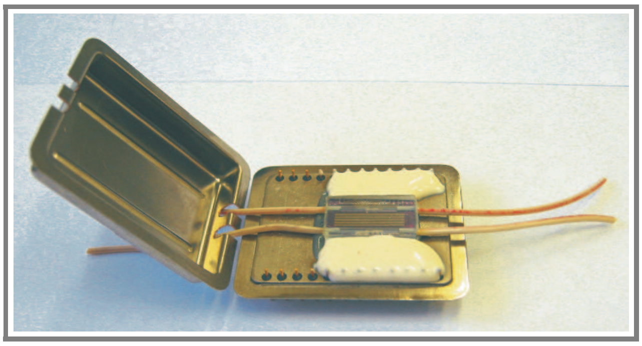

Fig. 4. TCD unit in an open package.

individual TCD silicon chips and these chips were subjected to anisotropic etching of the Si substrate through the windows opened previously in the double-layer dielectric mask (Fig. 2b). TMAH (tetra methyl ammonium hydroxide and water, $N(CH_3)_4OH + H_2O$) solution was used at 85°C. The duration of the etching process was 200 minutes. During this step SiO_2/Si_3N_4 mask layers were laterally under-etched and Pt resistors were suspended over the edges of grooves (Fig. 2c). Groove final dimensions are: width – 400 μm, depth – 100 μm, length – 15 mm. The side-walls correspond to the (111) crystal planes (Fig. 3). High lateral etch rate under the mask edge is caused by the presence of convex corners and high index crystal planes. Suspended resistors are extremely delicate, therefore even the steps of DI water rinsing and drying require special procedures to avoid mechanical damage. The glass plate was aligned and bonded with the silicon chip (Fig. 2d) and placed on the IC package platform. Channel outlets were connected to the external gas tubing and electrical wire connections were made between the TCD bonding pads and package pins (Fig. 4).

4. Results

Newly designed and fabricated TCD detectors are sensitive both to the gas flow velocity changes and gas mixture composition. In the chemical detection mode gas flow velocity should be very stable and each instability has to be compensated by the application of the reference flow channel. In the flow meter mode the gas type should be determined. Static, as well as dynamic, tests were done with the nitrogen gas and flow and electrical system set-up shown in Fig. 5.

Tests were done using a Keithley KPCI-3108 Series PCI Bus Data Acquisition Board and LabView 6.1 software installed on a standard PC. Two TCD units were used for dynamic tests – one of them was active, the second one was electrically passive and served to establish the same gas flow resistance, which is important for the gas flow switching. The active TCD unit was powered by a 8 $V_{(DC)}$ battery source. Flow switching from one channel to another was performed with an electrically activated Humphrey valve of the MINI_MYTE 41E1 type. A Tylan FC-260 mass flow controller was used as the reference, supported by an additional precise regulator.

Flow resistance versus flow rate characteristics are presented in Fig. 6. Points around the 0 ml/min value are distributed in a slightly non-symmetric way. This can be explained by the non-symmetric bending of the resistors inside the channel (Fig. 3). The U_{OUT} versus nitrogen gas flow rate characteristics (Fig. 7) are nonlinear in the 0–50 ml/min range. Nonlinearity seems to be low and sensitivity high in the flow region below 10 ml/min. It was also observed that in the case of TCD outlet opened to the ambient, the results close to 0 ml/min were disturbed by the penetration of air into the detector flow channel, and the resultant changes of the thermodynamic properties of the gas. Dynamic response times of the new TCD were measured in comparison to those of a piezoresistive pressure sensor (Fig. 8). Sampling frequency to analyze both output signal slopes was 10 000/s for a 3 s period test. Nitrogen flow was switched from 0 to 10 ml/min and back. The pressure sensor and TCD required 26.6 ms and 46.3 ms to reach 0% → 90% ΔU_{OUT}, respectively. Switching from 10 to 0 ml/min revealed slower response times of the pressure sensor (66.6 ms) and slightly faster TCD action (40.7 ms). Dynamic test results show that TCD units are much faster than the classic flow meter devices.

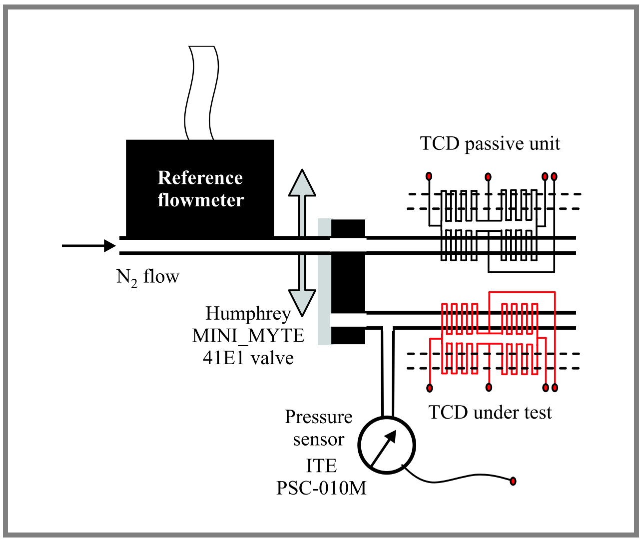

Fig. 5. Flow and electrical system used for static and dynamic tests of TCDs.

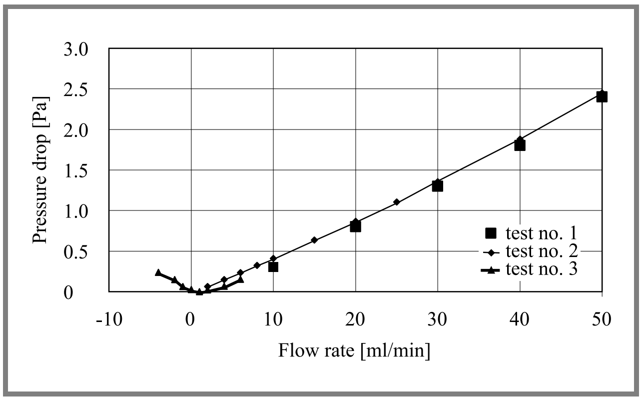

Fig. 6. Flow resistance of a TCD as a function of flow rate.

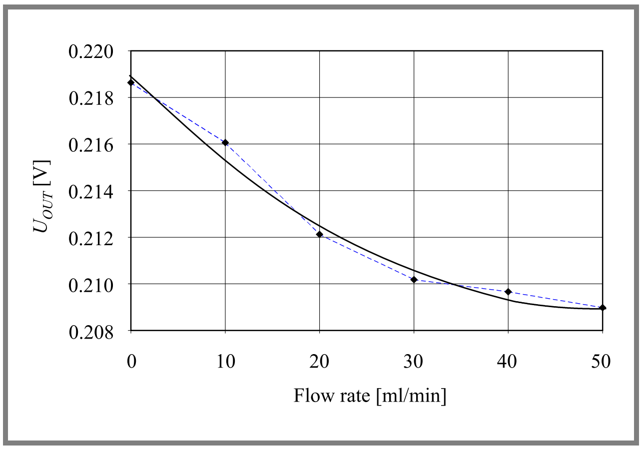

Fig. 7. Static U_{OUT} as a function of nitrogen flow rate.

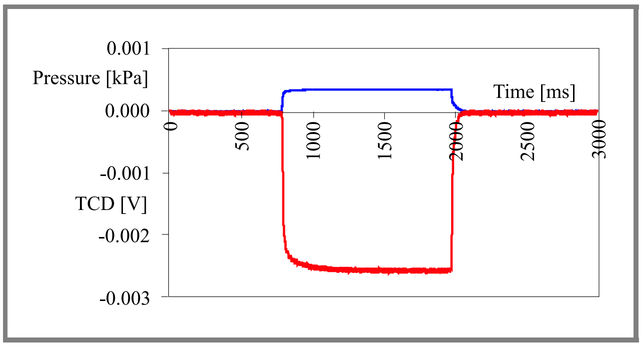

Fig. 8. Dynamic output of a TCD and a pressure sensor as a function of time. Switching of the nitrogen flow: 0 → 10 → 0 ml/min.

References

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