



US010189700B2

(12) **United States Patent**
Manos et al.

(10) **Patent No.:** **US 10,189,700 B2**(45) **Date of Patent:** **Jan. 29, 2019**

(54) **INTEGRATED CIRCUIT (IC) CHIP WITH A SELF-CONTAINED FLUID SENSOR AND METHOD OF MAKING THE CHIP**

(71) Applicant: **U.S.A. as represented by the Administrator of the National Aeronautics and Space Administration**, Washington, DC (US)

(72) Inventors: **George Manos**, Laurel, MD (US); **Manuel A. Balvin**, Springfield, VA (US); **Michael P. Callahan**, Boise, ID (US)

(73) Assignee: **The United States of America as represented by the Administrator of NASA**, Washington, DC (US)

(*) Notice: Subject to any disclaimer, the term of this patent is extended or adjusted under 35 U.S.C. 154(b) by 50 days.

(21) Appl. No.: **15/278,359**

(22) Filed: **Sep. 28, 2016**

(65) **Prior Publication Data**

US 2018/0086629 A1 Mar. 29, 2018

(51) **Int. Cl.**

G01F 1/708 (2006.01)
G01K 7/01 (2006.01)
B81B 7/00 (2006.01)
B81C 1/00 (2006.01)
G01F 1/684 (2006.01)

(52) **U.S. Cl.**

CPC **B81B 7/0061** (2013.01); **B81C 1/00309** (2013.01); **G01F 1/7084** (2013.01); **G01K**

7/01 (2013.01); **B81B 2201/0278** (2013.01); **G01F 1/6845** (2013.01)

(58) **Field of Classification Search**

CPC **B81B 7/00**; **B81B 7/006**; **B81B 7/0061**;
B81C 1/00; **B81C 1/003**; **B81C 1/003**;
B81C 1/00309; **G01F 1/70**; **G01F 1/78**;
G01F 1/7084
USPC **257/467**
See application file for complete search history.

(56) **References Cited**

U.S. PATENT DOCUMENTS

7,181,963 B2 * 2/2007 Bork G01F 1/68
73/204.26
7,259,080 B2 * 8/2007 Quenzer et al. H01L 21/46
438/455

* cited by examiner

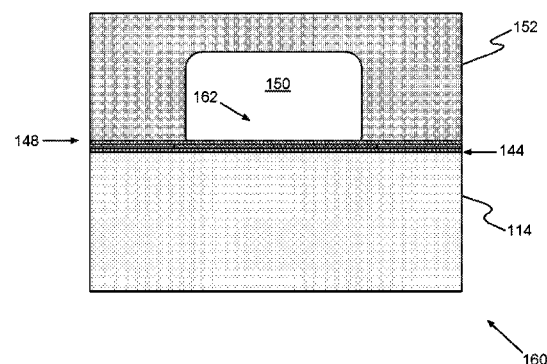
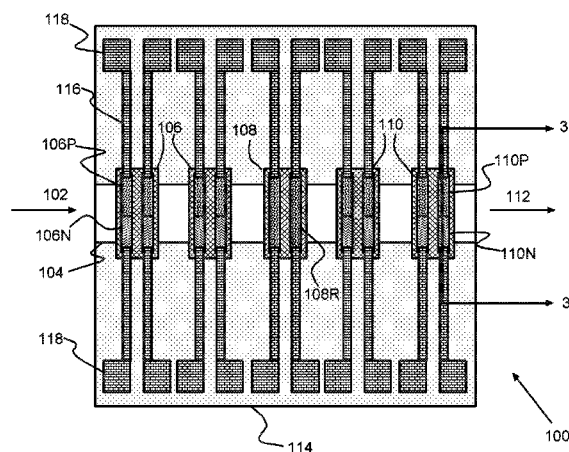
Primary Examiner — Monica D Harrison

(74) *Attorney, Agent, or Firm* — Christopher O. Edwards;
Bryan A. Geurts; Mark P. Dvorscak

(57) **ABSTRACT**

An integrated circuit (IC) chip with a self-contained fluid sensor and method of making the chip. The sensor is in a conduit formed between a semiconductor substrate and a non-conductive cap with fluid entry and exit points through the cap. The conduit may be entirely in the cap, in the substrate or in both. The conduit includes encased temperature sensors at both ends and a central encased heater. The temperature sensors may each include multiple encased diodes and the heater may include multiple encased resistors.

5 Claims, 5 Drawing Sheets



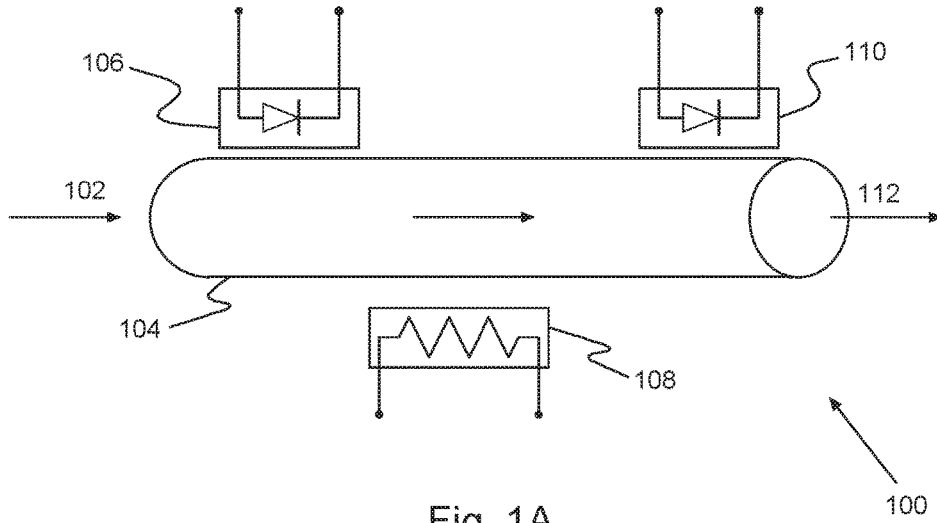


Fig. 1A

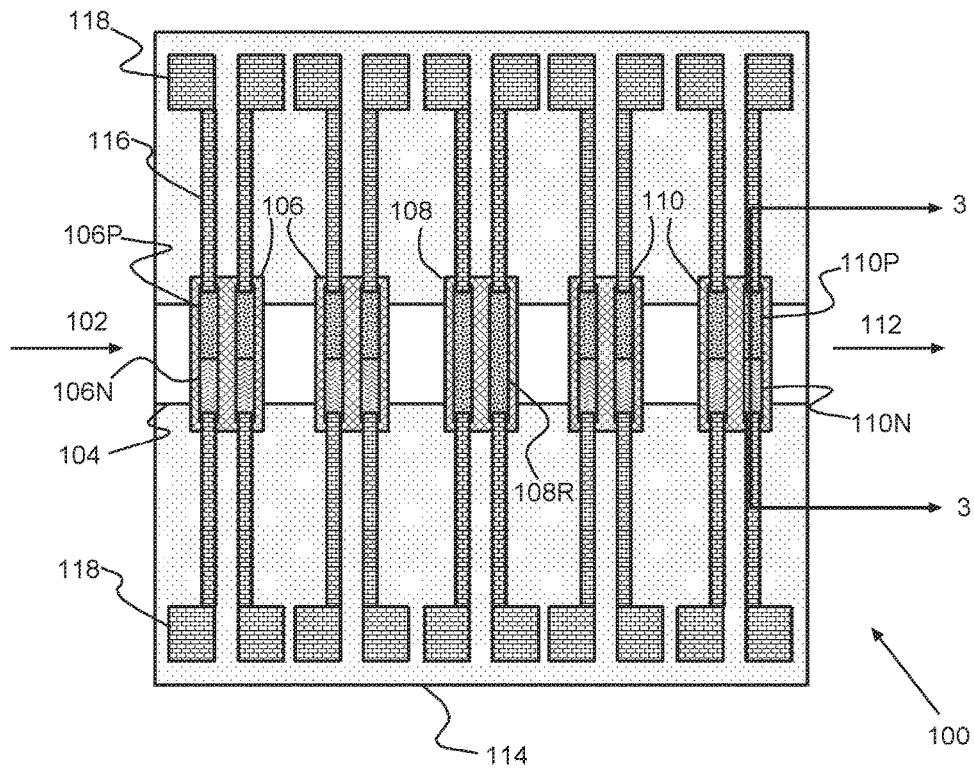


Fig. 1B

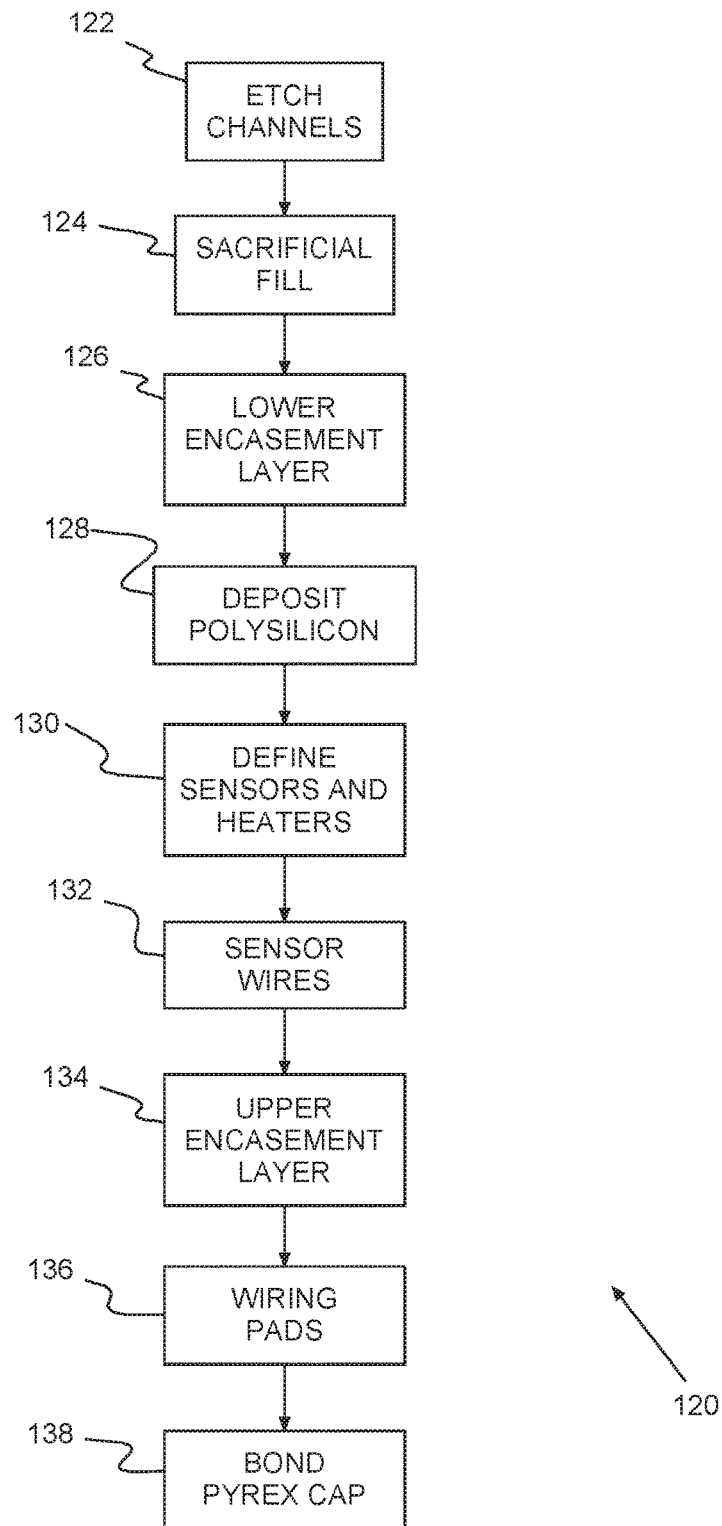


Fig. 2

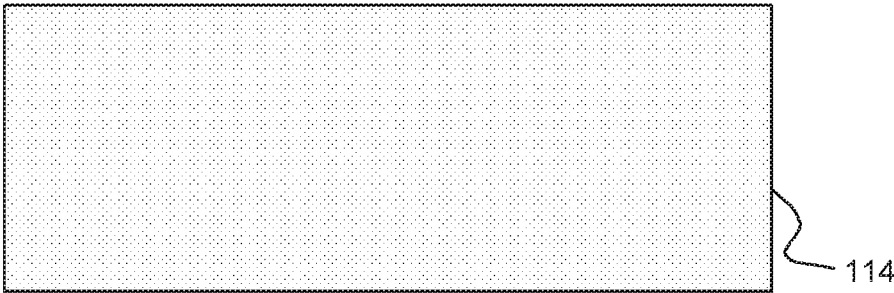


Fig. 3A

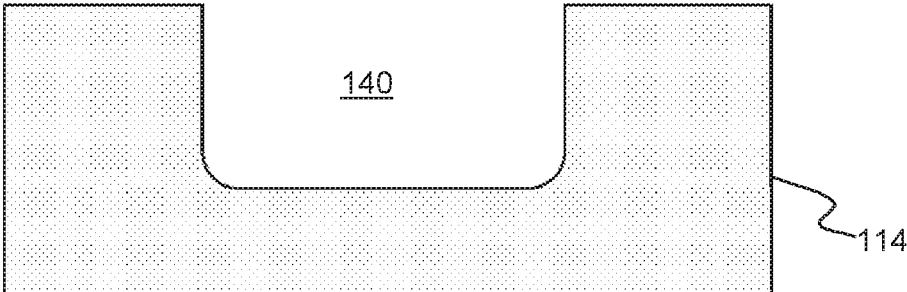


Fig. 3B

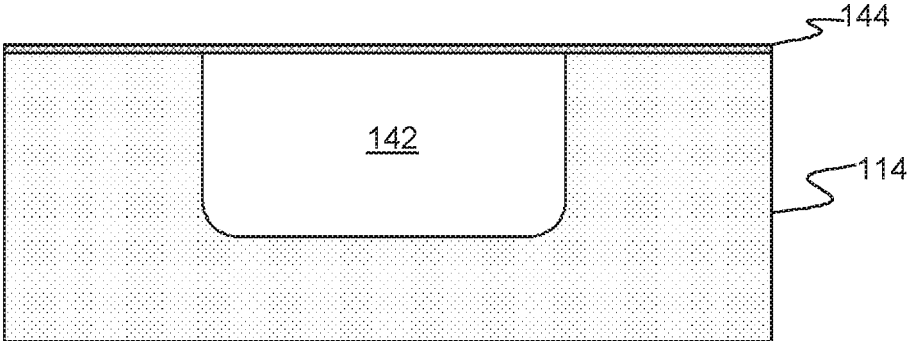


Fig. 3C

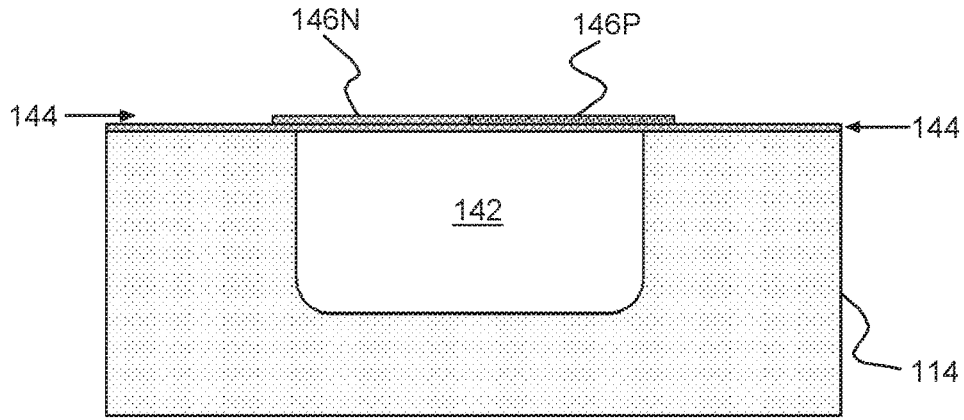


Fig. 3D

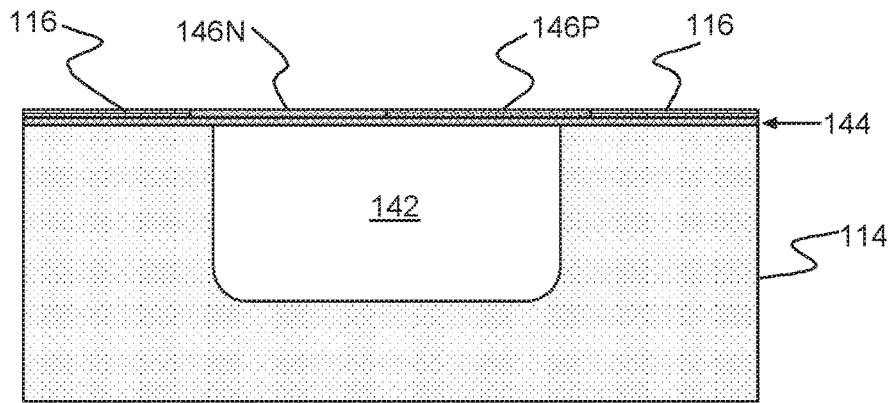


Fig. 3E

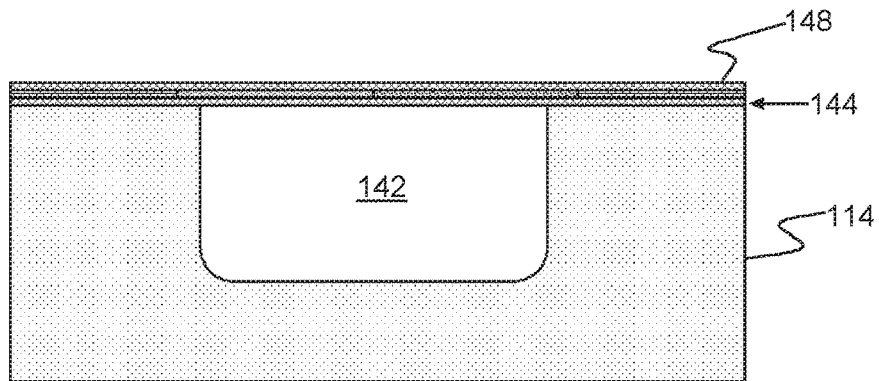


Fig. 3F

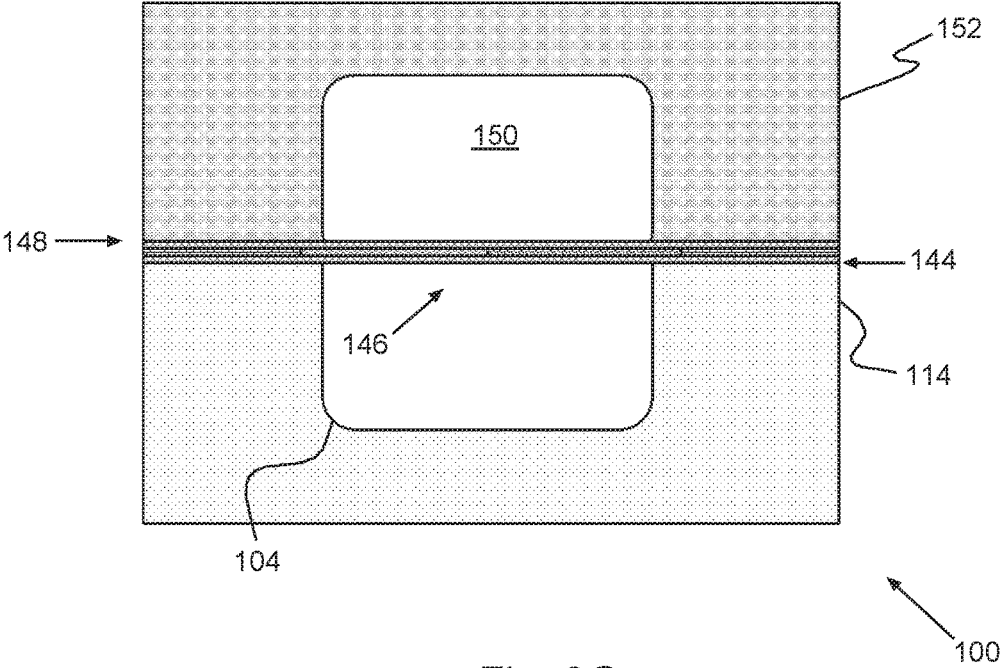


Fig. 3G

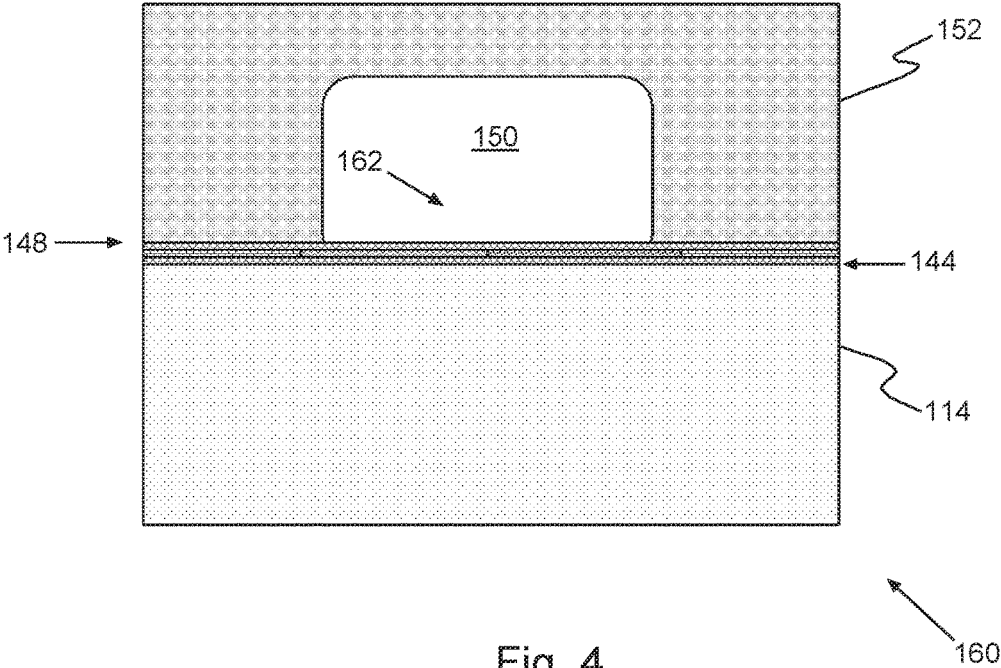


Fig. 4

INTEGRATED CIRCUIT (IC) CHIP WITH A SELF-CONTAINED FLUID SENSOR AND METHOD OF MAKING THE CHIP

ORIGIN OF THE INVENTION

The invention described herein was made by an employee of the United States Government, and may be manufactured and used by or for the Government for governmental purposes without the payment of any royalties thereon or therefor.

BACKGROUND OF THE INVENTION

Field of the Invention

The present invention is generally related to instruments for measuring fluid temperature and flow rate and more particularly to sensors for space-bourne applications for measuring fluid temperature and flow rate.

Background Description

National Aeronautics and Space Administration (NASA) scientists are developing next generation microfluidic instruments, e.g., lab-on-a-chip, for space based applications and on planetary bodies. Producing a lab-on-a-chip requires advancements in miniaturization, especially for analytical systems, requires an on-chip, in situ fluid flow and fluid temperature measurement capability. Unfortunately, state of the art sensors are either too large for integration on-chip, requiring diverting fluid off-chip for measurement by a flow sensor and a temperature sensor. Only after the measurement is complete is the fluid returned to the chip. These sensors are still incapable of measuring flow rates, while requiring increased fluid volume (and corresponding weight) at a higher risk of on-board system fluid leaks.

Thus, there is a need for an on-chip flow and temperature sensor for measuring microfluidic flow rates and temperatures, and more particularly, for accurately measuring microfluidic flow rates and temperatures for next generation microfluidic space-bourne instruments traveling to extraterrestrial planetary bodies.

SUMMARY OF THE INVENTION

An aspect of the invention is a self-contained fluid flow rate and temperature sensor;

Another aspect of the invention is an on-chip capability for measuring nano-liter per minute fluid flow rates over a broad temperature range;

It is yet another aspect of the invention an on-chip, self-contained fluid flow rate and temperature sensor for measuring fluid flow at rates in the nano-liter per minute range and over temperatures ranging from below -80°C . to above 150°C . in any application that requires accurate temperature and flow measurements.

The present invention relates to an integrated circuit (IC) chip with a self-contained fluid sensor and method of making the chip. The sensor is in a conduit formed between a semiconductor substrate and a non-conductive cap with fluid entry and exit points through the cap. The conduit may be entirely in the cap, in the substrate or in both. The conduit includes encased temperature sensors at both ends and a central encased heater. The temperature sensors may each include multiple encased diodes and the heater may include multiple encased resistors.

BRIEF DESCRIPTION OF THE DRAWINGS

The foregoing and other objects, aspects and advantages will be better understood from the following detailed description of a preferred embodiment of the invention with reference to the drawings, in which:

FIGS. 1A-B show a schematic and plan view example of a Dynamic On-Chip Flow and Temperature Sensor (DOC-FlaTS) for sensing on-chip fluid flow and temperature, according to a preferred embodiment of the present invention;

FIG. 2 shows a flow diagram for fabricating the sensors on a semiconductor wafer;

FIGS. 3A-G show a cross-sectional example of forming an IC chip with a preferred flow and temperature sensor;

FIG. 4 shows an alternate embodiment cross-sectional example of forming an IC chip with a preferred flow and temperature sensor.

DESCRIPTION OF PREFERRED EMBODIMENTS

Turning now to the drawings, and more particularly, FIGS. 1A-B show a schematic and plan view example of a Dynamic On-Chip Flow and Temperature Sensor (DOC-FlaTS) **100** for sensing on-chip fluid flow and temperature, according to a preferred embodiment of the present invention. Preferred sensors **100** provide on-board, on-chip capability for measuring flow rates in the nano-liter per minute (nl/min) range, at temperatures from below -80°C . to above 150°C . in any application that requires accurate temperature and flow measurements.

Fluid enters **102** at one end of an embedded conduit **104** where one or more front-end or input temperature sensors **106** measure fluid temperature. One or more heating elements **108** selectively heat the fluid before it passes by one or more back-end or output temperature sensors **110** to exit **112** the conduit **104**. Preferably, the channel **102** is formed partially in, or on, a capped semiconductor, e.g., silicon (Si), wafer **114**. Preferably also, temperature sensors **106**, **110** and heater **108** are encased in Silicon Nitride (SiN) for electrical isolation, and are connected through wires **116** externally at pads **118**. For maximum fluid surface contact, preferably, the temperature sensors **106**, **110** are suspended in the conduit **104** to optimize sensitivity and response time.

In this example the input temperature sensors **106** and output temperature sensors **110** each include two pair, or 4, polysilicon diodes, i.e., PN junctions **106P**, **106N** and **110P**, **110N**. Also in this example, the heaters **108** are a pair of polysilicon resistors (R) **108R** with resistor current (I_R) selectively applied to dissipate power (I_R^2R) generating localized heat in the conduit **104**. The polysilicon diodes **106P**, **106N** and **110P**, **110N** exhibit determinable sensitivity to junction temperature (T). Diode **106P**, **106N** and **110P**, **110N** junction thermal voltage (V_T) is a constant at any absolute temperature, defined by $V_T=kTq$, where k is the Boltzmann constant, q is the magnitude of charge of an electron, and the rate of change is ~ 2.2 millivolts (~ 2.2 mV) per degree Kelvin ($^{\circ}\text{K}$).

Fluid entering **102** the conduit **104** at input temperature sensors **106** exhibits a temperature difference (dT) with the fluid exiting **112** at output temperature sensors **110**, that may also be measured among individual sensor junctions **106P**, **106N** and **110P**, **110N**. That temperature difference is dependent upon the length of time passing heaters **108** and between sensors **106**, **110**, and, therefore, is a measure of fluid flow or velocity through the conduit **104**. During

3

operation, temperature sensors **106**, for example, **110** may be biased at a fixed current (I_D) or fixed voltage (V_D) and absolute temperature (T) for each diode may be derived from the well-known, ideal diode relationship,

$$I_D = I_S \left(e^{\frac{V_D}{nV_T}} - 1 \right),$$

where n is a diode quality factor.

Thus, the front-end temperature sensors **106** allow for one or more initial fluid temperature readings before passing the heater **108**. Fluid thermal conductance, fluid heat capacity combine with heat from power to the heaters **108** to elevate temperature of the fluid traversing the conduit **104**. The back-end temperature sensors **110** allow for measuring heat injected into the conduit **104** fluid from dT . For example, the temperature difference reflected in diode voltage change (dV_D) between sensor junctions **106P**, **106N** and **110P**, **110N** provides a measure of microfluidic flow rate through the conduit **104** according to the relationship, $dV_D/dT = V_D/T - E_g/T - 2Kq$, where E_g is the substrate energy band gap. Accordingly, a self-contained preferred sensor **100** provides a single chip solution for accurately measuring microfluidic, on-chip flow rates and temperatures, especially for next generation microfluidic space-borne instruments.

FIG. 2 shows a flow diagram **120** example of fabricating the preferred sensors (**100** in FIGS. 1A-B) on a semiconductor substrate wafer **114**. First in this example, micro-channels **104** are etched **122** into the wafer. A sacrificial layer is deposited **124** on to the wafer to back fill the substrate channels **104** and re-planarize to the wafer surface. A lower encasement layer is formed **126** on the wafer, e.g., using low pressure chemical vapor deposition (LPCVD). Polysilicon, doped or undoped, is deposited **128** on the lower encasement layer, patterned and selectively doped, to define **130** the temperature sensors **106**, **110**, e.g., diodes, and resistive heater(s) **108**. Connective wiring is formed **132** to each of the temperature sensor **106**, **110** diodes and the resistive heater(s) **108**. Forming **134** an upper encasement layer on the patterned polysilicon, encases and encloses all temperature sensors **106**, **110** and heaters **108**. Pads for probing or connecting to other sensing circuits are formed **136**, e.g., at the edge of the sensor **100**, wafer **114** or each chip, and wafer and/or chip fabrication may complete. A corresponding micro-channel in a non-conductive cap, e.g., pyrex, bonded **138** to the wafer **114** forms/completes the conduit **104**.

FIGS. 3A-G show a cross-sectional example of forming an IC chip with a preferred flow and temperature sensor **100** through **3-3** in FIG. 1B, formed as described in FIG. 2 and with like features labeled identically. Although the channel **140** in silicon wafer **114** shown in this example has a rectangular cross-section with rounded corners, this is for example only. The channel **140** may have any shape as desired formed in the substrate **114**, e.g., completely rounded or cylindrical, oval or square, as selected by design and constrained by fabrication.

Typically, a preferred sensor **100** occupies a small portion of the overall integrated circuit (IC) chip. For a 10 mm×15 mm chip, for example, the flow and temperature sensor **100** may be 0.08 mm×0.8 mm within a 10.0 mm long by 0.08 mm wide channel **140** etched **122** in the semiconductor wafer **114**. A Low Thermal Oxide (LTO) may be deposited **124** to form sacrificial material, back filling the channels **140**, e.g., using LPCVD. Then, sacrificial material is

4

removed from the wafer, e.g., using a typical chemical-mechanical (chem-mech) polish, to re-planarize the wafer surface, with the back fill LPCVD LTO **142** remaining in the channel(s) **104** and providing a flat surface for subsequent fabrication.

Having filled and re-planarized, LPCVD SiN layer forms **126** the lower encasement layer **144**. The lower encasement layer **144** is 100-200 nanometers thick (100-200 nm), preferably, 150 nm thick. The lower encasement layer **144** is patterned, e.g., using a typical photolithographic pattern (mask) and etching, to define sensor **106**, **110** locations and heater **108** locations. The patterned lower encasement layer **144** positions the temperature sensor **106**, **110** diodes and resistive heaters **108** in the conduit **104**, e.g., centrally located, for maximum fluid surface contact in the conduit **104**.

Eighty micron (80 μ m) long polysilicon strips, preferably, 0.2 μ m thick and 0.5-1.0 μ m wide, are formed using a LPCVD and mask and etch. The polysilicon strips locate the temperature sensor **106**, **110** diodes and heater resistors **108R**. For example, a 0.2 μ m thick polysilicon layer may be deposited as doped polysilicon, e.g., P-doped, undoped, or intrinsic polysilicon. Patterning the polysilicon defines the strips, and doping the strips defines diodes and heaters. For undoped polysilicon, for example, the strips may be implanted with P-type dopant, preferably, boron. Masking the P-doped polysilicon to protect the resistors **108R** and portions of the diode strips **146P**, exposed portions **146N** of the strips are implanted with N-type dopant, preferably, phosphorus, forming polysilicon diodes **146**.

After forming **132** connective wires **116** to each of the temperature sensor **106**, **110** diodes and the resistive heater(s) **108**, an upper encasement layer **148** is deposited. Preferably, the upper encasement layer **148** is SiN, 50-150 nm, and preferably, 150 nm thick. Then, the upper encasement layer **148** is patterned **134**, encasing and enclosing all polysilicon temperature sensors **106**, **110** and heaters **108** in a silicon nitride membrane strong enough to withstand the laminar flow within the conduit **104**. Other IC circuits, e.g., sensing and support circuits, may be formed tailored by design to the preferred sensors **100**. Bond pads may be formed **136**, for example, by depositing and patterning a suitable metal with the bond pads **118** remaining exposed. The bond pads **118** provide, for example, for connection to another chip, for wafer level test probing, or for connection to other sensing circuits, e.g., at the edge of the sensor.

In this example IC fabrication completes **138** connecting circuits together, to bond pads **118** and off chip, and the remaining LPCVD LTO **140'** is removed suspending the temperature sensors **106**, **110** and heaters **108** in the middle of the conduit **104**. In operation the suspended temperature sensors **106**, **110** are completely immersed in the fluid in the conduit **104**, thermally isolating them from ambient for faster flow and temperature change responses.

After completing ICs **138**, bonding a corresponding channel **150** in a pyrex cap **152** to the wafer **114** completes the conduit **104**. The bonded pyrex cap **152** houses the flow and temperature sensors in the completed conduit **104**. The pyrex cap **152** also houses a tapered inlet via **102** and outlet via **112** clamped to the wafer **114** with nano-ferrules and capillary tubes (not shown) to produce a leak-tight channel to macro-scale connection.

FIG. 4 shows an alternate embodiment cross-sectional example of forming an IC chip with a preferred flow and temperature sensor **160**, also formed as described in FIG. 2 and with like features labeled identically. In this alternate example channels are not formed in the wafer **114**. Instead,

5

the lower encasement layer **144** is formed on the semiconductor wafer **114**, placing the sensors **162** and heaters at the bottom of the conduit **104**. The upper encasement layer **148** encases and encloses all polysilicon sensor diodes **162** and heaters. Also in this example, the channel **150** in the pyrex cap **152** bonded to the wafer **114** forms the entire sensor conduit **104**. While this embodiment is less thermally isolated, on the bottom on the conduit **104**, the temperature sensors **106**, **110** and heaters **108** are more mechanically robust and simpler to fabricate.

Advantageously, preferred on-chip flow and temperature sensors eliminate the need to route fluid off-chip and out of an analytical system for separately measuring flow rate and temperature. Preferred sensors are capable of accurately measuring flow rates down to the nano-liter per minute range. Temperature measurement capabilities range from below -80°C . to above 150°C . Further, preferred sensors provide for analyzing relatively small, even minute, individual system samples, thereby reducing the total required fluid mass and volume. This reduction is a key requirement for space-borne microfluidic instruments used on both space and planetary platforms. Thus, preferred sensors provide an on-board, on-chip application capability wherever accurate temperature and flow measurements may be required.

While the invention has been described in terms of preferred embodiments, those skilled in the art will recognize that the invention can be practiced with modification within the spirit and scope of the appended claims. It is intended that all such variations and modifications fall within the scope of the appended claims. Examples and drawings are, accordingly, to be regarded as illustrative rather than restrictive.

6

What is claimed is:

1. An integrated circuit (IC) chip including a self-contained fluid sensor comprising:
 - a semiconductor substrate;
 - a non-conductive cap disposed on said semiconductor substrate;
 - a conduit at the interface between said semiconductor substrate and said nonconductive cap, each end of said conduit passing through said non-conductive cap;
 - a temperature sensor in said conduit at said each end; and
 - a heating element in said conduit between the temperature sensors wherein the temperature sensors and said heating element are encased in dielectric; further wherein said non-conductive cap is a pyrex cap with a channel at said semiconductor substrate, wherein said conduit comprises said channel and
 wherein said semiconductor substrate comprises a micro-channel, said conduit further comprises said micro-channel, the encased temperature sensors and said heating element being suspended in said conduit at said interface.
2. An IC chip as in claim 1, wherein the encased temperature sensors and the encased heating element are disposed on said semiconductor substrate at said interface.
3. An IC chip as in claim 1, wherein said semiconductor substrate is silicon and each said temperature sensor comprises one or more polysilicon diodes and said heating element comprises one or more polysilicon resistor.
4. An IC chip as in claim 3, wherein said one or more polysilicon diodes comprises two pair of diodes and said polysilicon resistor comprises a pair of resistors.
5. An IC chip as in claim 4, further including one or more circuits on said silicon substrate and connected to said two pair of diodes and said pair of resistors.

* * * * *