

Microcantilever Heater-Thermometer with Integrated Temperature-Compensated Strain Sensor

CROSS REFERENCE TO RELATED APPLICATION

[001] Not Applicable.

STATEMENT REGARDING FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

[002] This invention was made with government support under Subcontract No. B552749 to the Regents of the University of California and Georgia Tech Research Corporation under United States Government Prime Contract No. W-7405-RNG-48 represented by the Department of Energy National Nuclear Security Administration (DOE/NNSA) for the management and operation of Lawrence Livermore National Laboratory (LLNL). The government has certain rights in the invention.

BACKGROUND OF THE INVENTION

[003] This invention is in the field of atomic force microscopes and microcantilevers. This invention relates generally to a microcantilever having a temperature compensating piezoresistive strain sensor and integrated heater-thermometer. This invention also relates to methods of using such a cantilever in the fields of thermodynamic measurements and chemical/biochemical sensing.

[004] Since the invention of the atomic force microscope (AFM), microcantilevers have become one of the most frequently used microelectromechanical systems (MEMS) devices with applications ranging from scanning probe microscopy to bio/chemical sensing. Microcantilevers are often functionalized by introducing current traces, piezoresistive or piezoelectric materials to realize specific applications. Heatable microcantilevers having either doped single-crystalline or polycrystalline

silicon or patterned metal traces allow current flow so that they can be heated by means of resistive (Joule) heating. This heating in microcantilevers can be used for bimorph actuation, thermomechanical data storage, thermal displacement sensing in contact and tapping modes, novel nano-material synthesis, nanoscale thermal analysis and nanoscale thermal manufacturing. Recently, cantilever type micro-hotplates have been reported as an alternative platform for calorimetry. A cantilever type micro-hotplate fabricated based on silicon technology has been introduced and several microcantilever hotplates were designed, fabricated, and characterized to investigate response time and temperature uniformity.

[005] Microcantilevers with integrated piezoresistive strain sensors are mainly used to replace optical deflection sensing but are also employed in various sensing applications such as gas flow sensing, acceleration sensing, microjet measurements and bio/chemical sensing. Especially as bio/chemical sensors, piezoresistive microcantilevers are often prepared with a selective coating sensitive to a specific analyte. Analyte adsorption induces static deflection by creating a surface stress, and thus embedded piezoresistors can measure analyte adsorption.

[006] Microcantilevers having both resistive heaters and piezoresistors can offer simultaneous heating and deflection sensing. These hybrid types have been used as multi-functional scanning probes in thermomechanical data storage. Similarly, microcantilevers with the ability of independent heating and sensing operation that have high sensitivity to surface stress could be used for a variety of sensor applications. One example would be calorimetry of a material adhered to the cantilever surface. Chemical processes such as melting and evaporation and chemical reactions between substances could be triggered by the heaters while the changes in the surface stresses on the cantilever are monitored and can give information about the material or reaction properties. Other examples include biochemical sensing, where one might wish to interrogate the temperature-dependence of biochemical binding to a microcantilever.

[007] One strategy for suppressing unwanted signals, such as temperature drift, in piezoresistive cantilever sensors is to fabricate cantilever pairs for a differential measurement. Two microcantilevers with identical piezoresistive strain gauges can be arrayed closely and interfaced in a Wheatstone bridge to cancel temperature drift with the assumption they have the same temperature progression. However, this approach would not be appropriate for cases in which the two cantilevers experience different temperatures. This could be the case when a reactive coating modifies the thermal properties of one cantilever. Temperature deviations between the two devices can also be caused by the system environment, *e.g.* by thermal gradients due to gas flow directions. On-chip temperature compensation for piezoresistive cantilever sensors has been demonstrated, but these cantilevers did not have integrated heater-thermometers. Furthermore, all previous approaches to on-chip temperature compensation use the principle of a Wheatstone bridge circuit on the cantilever, a method which assumes unidirectional, equal stress in all resistors. However, for chemical sensing, in which a reactive layer causes a surface stress on the silicon surface, the stress distribution in the cantilever is complex and three-dimensional. Therefore, it is favorable to incorporate independent sensors for stress and temperature in the cantilever to correct the effect of thermal variations on the mechanical signal.

SUMMARY OF THE INVENTION

[008] The present invention provides microcantilever hotplate devices which incorporate temperature compensating strain sensors into a cantilever structure. The devices of the present invention comprise microcantilevers having temperature compensating strain sensors and resistive heaters. In an embodiment, the devices of the present invention are useful as microhotplates and are capable of heating a material, compound, or species which is bound to or provided on the microhotplate surface. The present invention also provides methods for using a microcantilever hotplate for temperature

compensated surface stress measurements, chemical/biochemical sensing, measuring various properties of compounds adhered to the microcantilever hotplate surface, or for temperature compensated deflection measurements.

[009] In an exemplary embodiment, a microhotplate of the present invention comprises a cantilever having a fixed end and a free end; a pair of piezoresistive sensors integrated into the cantilever near the fixed end, wherein the first piezoresistive sensor is aligned along a first crystal axis of the cantilever and has a first piezoresistive coefficient, and wherein the second piezoresistive sensor is aligned along a second crystal axis of the cantilever and has a second piezoresistive coefficient that is less than said first piezoresistive coefficient; and a heater-thermometer integrated into the cantilever. In a preferred embodiment the second piezoresistive coefficient is very small (*i.e.* less than or equal to 1% of the first piezoresistive coefficient). As used herein, the expression “piezoresistive sensors integrated into the cantilever near the fixed end” refers to a relative position of the piezoresistive sensors between 0 and 200 μm of the cantilever fixed end, preferably for some applications between 0 and 50 μm of the fixed end of the cantilever. This expression also includes embodiments where the piezoresistive sensors are spatially coincident with the fixed end of the cantilever.

[010] In an embodiment preferred for some applications, the present invention provides a method of sensing a surface stress of a microhotplate. A method of this aspect comprises the steps of providing a microhotplate of the present invention having a pair of piezoresistive sensors, electrically connecting the first and second piezoresistive sensors in a Wheatstone bridge circuit or other circuit capable of sensing a change in a resistance, and sensing a change in a resistance of one of the piezoresistive sensors, thereby sensing a surface stress in the microhotplate. In a preferred embodiment of this method, the piezoresistive sensors are electrically connected in the Wheatstone bridge circuit or resistance sensing circuit in an arrangement which compensates for a change in the resistances of the piezoresistive sensors due to temperature. In another preferred embodiment, the

piezoresistive sensors are positioned near the fixed end of a cantilever of the microhotplate in proximity to each other, such that they have a substantially identical temperature. In some embodiments the first and second piezoresistive sensors are positioned less than 10 μm , 25 μm , 50 μm , 100 μm , or 250 μm from one another

[011] In another embodiment preferred for some applications, the present invention provides a method of sensing a property of a compound, species, or substance. A method of this aspect comprises the steps of providing a microhotplate of the present invention having a pair of piezoresistive sensors positioned near the fixed end of a microcantilever and a heater-thermometer positioned in the free end of the microcantilever; contacting the free end of the microcantilever with a compound, species, or substance; providing a voltage or current to the heater-thermometer to affect a temperature change in the microcantilever and compound, species, or substance and wherein such a temperature change produces a surface stress in the microcantilever; and sensing a change in a resistance of at least one of the piezoresistive sensors. This method may also further comprise sensing the resistance of the heater-thermometer to allow for determination of the temperature of the cantilever and compound, species, or substance. In an embodiment, the change in resistance of a piezoresistive sensor is sensed by electrically connecting one or both piezoresistive sensors in a Wheatstone bridge circuit, for example in a manner to compensate for a change in the resistances of the piezoresistive sensors due to temperature.

[012] Useful properties that are capable of being sensed by the methods of the present invention include, but are not limited to: a melting point of the compound, species, or substance; a boiling point of the compound, species, or substance; a binding energy of the compound, species, or substance; a heat capacity of the compound, species, or substance; a mechanical expansion of the compound, species, or substance; a mechanical contraction of the compound, species or substance; or other properties. In some embodiments, a surface stress produced in the microcantilever may be

produced by a specific event occurring or at a specific temperature. Such events include, but are not limited to: a change in the state of the compound, species, or substance, for example melting or boiling; a reduction in the amount of the compound, species, or substance present on the microcantilever, for example caused by evaporation; a change in the extent of binding of the compound, species or substance to the microcantilever, for example a reduction in binding from 100% of the initial amount of compound, species or substance bound to a percentage less than 100% of the initial amount bound; a mechanical expansion of the compound, species, or substance; a mechanical contraction of the compound, species or substance; or other events.

[013] In an alternative embodiment, the present invention provides a method for sensing a species bound to a microhotplate. A method of this aspect comprises the steps of: providing a first microhotplate of the present invention having a pair of piezoresistive sensors positioned near the fixed end of a microcantilever and a heater-thermometer positioned in the free end of the microcantilever; providing a species capable of binding to the free end of the cantilever, wherein the binding of the species to the cantilever produces a surface stress in the cantilever; and sensing a change in a resistance of at least one of the piezoresistive sensors, thereby sensing the species bound to the microhotplate. The method may further comprise the step of providing a voltage or current to the heater-thermometer to affect a change in the temperature of or fix the temperature of the microcantilever. In a preferred embodiment, the free end of the cantilever comprises a substance capable of selectively binding the species of interest. In a preferred embodiment, the change in resistance of a piezoresistive sensor is sensed by a method comprising electrically connecting one or both piezoresistive sensors in a Wheatstone bridge circuit, preferably in a manner to compensate for a change in resistance of the piezoresistive sensors due to temperature.

[014] In another embodiment, the method of sensing a species bound to a microhotplate may further comprise the steps of: providing a second

microhotplate of the present invention substantially identical to the first microhotplate; providing the second microhotplate in an environment substantially identical to that of the first microhotplate, wherein the environment of the second microhotplate does not have the species of interest present; and electrically connecting the piezoresistive sensors of the first and second microhotplates in a Wheatstone bridge circuit in a manner that compensates for changes in resistance of the piezoresistive sensors due to effects other than binding of the species to the microcantilever.

[015] The present invention also provides a method of performing a temperature compensated deflection measurement. A method of this aspect comprises the steps of: providing a microhotplate of the present invention having a pair of piezoresistive sensors positioned near the fixed end of a microcantilever and a heater-thermometer positioned in the free end of the microcantilever; electrically connecting the piezoresistive sensors in a Wheatstone bridge circuit in a manner to compensate for a change in the resistance of the piezoresistive sensors due to temperature; and sensing a change in the resistance of at least one of the piezoresistive sensors due to a deflection of the free end of the microcantilever. This method may further comprise providing a voltage or current to the heater-thermometer to change or fix the temperature of the microcantilever.

[016] Without wishing to be bound by any particular theory, there can be discussion herein of beliefs or understandings of underlying principles relating to the invention. It is recognized that regardless of the ultimate correctness of any mechanistic explanation or hypothesis, an embodiment of the invention can nonetheless be operative and useful.

BRIEF DESCRIPTION OF THE DRAWINGS

[017] Figure 1 shows a schematic of two types of microcantilever hotplates with temperature-compensated piezoresistors.

[018] Figure 2 shows a schematic of six major fabrication processes to make the microcantilever hotplates with temperature-compensated piezoresistors.

[019] Figure 3 shows scanning electron micrographs of the fabricated microcantilever hotplates.

[020] Figure 4 shows simulated boron concentration using a process simulation tool for low and high doping processes.

[021] Figure 5 shows a testing circuit for electrical characterization of the microcantilever hotplates.

[022] Figure 6 shows data illustrating (a) Heater resistance and (b) heater power vs. applied voltage to the heater for two cantilever types; (c) Piezoresistor resistance and (d) piezoresistor power vs. applied voltage to the piezoresistors in $\langle 110 \rangle$ and $\langle 100 \rangle$ direction for two cantilever types.

[023] Figure 7 shows data illustrating the hot spot temperature of each device type for five different power levels from laser Raman thermometry.

[024] Figure 8 shows measurements of local temperature vs. relative position for type **A** and **B** (a) along the cantilever length, and (b) along the cantilever width.

[025] Figure 9 shows two circuit configurations using the on-chip resistors in the Wheatstone bridge.

[026] Figure 10 shows measured characteristics of the various resistors from a type **B** cantilever.

[027] Figure 11 shows a summary of cantilever deflection sensitivity up to 200 °C.

DETAILED DESCRIPTION OF THE INVENTION

[028] In general the terms and phrases used herein have their art-recognized meaning, which can be found by reference to standard texts, journal references and contexts known to those skilled in the art. The following definitions are provided to clarify their specific use in the context of the invention.

[029] “Heater-thermometer” refers to a resistive material which is capable of both generating heat and use as a means for measuring temperature. In an embodiment, a heater-thermometer is a thermistor. An ideal heater-thermometer is a material that has a resistance which is temperature dependant. Providing a current or voltage to a heater-thermometer can result in an increase in the temperature of the heater-thermometer through resistive (Joule) heating. Since the resistance of a heater-thermometer is temperature dependent, it can be used as means for measuring the temperature; *i.e.*, by measuring the resistance of the heater-thermometer, the temperature can be determined. A heater-thermometer useful with some embodiments of the present invention comprises doped silicon, for example silicon doped with phosphorus or boron.

[030] “Piezoresistive sensor” refers to a resistive material having resistance which changes when a strain is induced in the material, for example when the material is stretched or compressed. A piezoresistive sensor useful with some embodiments of the present invention comprises doped silicon, for example silicon doped with phosphorus or boron. In some embodiments of the present invention, piezoresistive sensors are integrated into a cantilever near the fixed end, and are useful for sensing surface stresses induced in the cantilever. According to this aspect, when a surface stress is induced in a cantilever which has an integrated piezoresistive sensor, the resistance of the piezoresistive sensor will change and can be sensed by a resistance sensing circuit thereby sensing the surface stress of the cantilever.

[031] “Wheatstone bridge” refers to an electric circuit which is capable of sensing a change in a resistance of an element of an electric circuit and/or

capable of determining an unknown resistance of an element of an electric circuit. Generally, a Wheatstone bridge circuit is constructed by positioning four resistors in a bridge circuit, where one the resistance of one resistor is unknown and the resistance of another resistor is finely adjustable. Figure 9 shows construction of two different embodiments of Wheatstone bridge circuits useful in the present invention. Figure 9 (a) depicts a Wheatstone bridge circuit in which the resistance of a piezoresistive sensor, for example positioned along a $\langle 110 \rangle$ or $\langle 100 \rangle$ crystal axis, is being sensed; Figure 9 (b) depicts a Wheatstone bridge circuit in which the resistance of a piezoresistive sensor positioned along a $\langle 110 \rangle$ crystal axis is being sensed in reference to a piezoresistive sensor positioned along a $\langle 100 \rangle$ crystal axis. The Wheatstone bridge circuit embodied in Figure 9 (b) is useful for compensating for the change of resistance of piezoresistive sensors due to temperature and determining the change of resistance of a piezoresistive sensor due to strain. A Wheatstone bridge similar to that of Figure 9 (b) can also be used for compensating for changes in resistance due to other effects.

[032] “Cantilever” and “microcantilever” are used interchangeably herein and refer to a structure having one fixed or attached end and one free or unattached end, for example a cantilever of an atomic force microscope. In some embodiments, the cantilevers of the present invention have dimensions on the order of 10 to 1000 μm .

[033] “Microhotplate” refers to a microcantilever having a resistive heater or heater-thermometer integrated into at least a portion of the free end of the microcantilever such that the microcantilever can be used to heat a material, species, or compound bound to or positioned on the microcantilever free end. A microhotplate is preferred to have a uniform temperature distribution across the free end of the microcantilever, and may have one or more resistive heater portions. The terms “microhotplate” and “microcantilever hotplate” are used interchangeably herein.

[034] “Thermal communication” refers to an orientation or position of two elements, such as a heater-thermometer and a piezoresistive sensor, such that there is more efficient transfer of heat between the two elements than if isolated or thermally insulated. Elements may be considered in thermal communication if heat is transported between one element and another more quickly than if the elements were isolated or thermally insulated. Two elements in thermal communication may reach thermal equilibrium and in some embodiments may be considered to be constantly at thermal equilibrium with one another.

[035] “Substantially identical” refers to two objects, values, or other items being the same or nearly the same.

[036] The microhotplate devices of the present invention comprise a cantilever having a fixed end and a free end; a pair of piezoresistive sensors integrated into the cantilever near the fixed end, wherein the first piezoresistive sensor is aligned along a first crystal axis of the cantilever and has a first piezoresistive coefficient, and wherein the second piezoresistive sensor is aligned along a second crystal axis of the cantilever and has a second piezoresistive coefficient that is less than the first piezoresistive coefficient; and a heater-thermometer integrated into the cantilever.

[037] In a preferred embodiment, the first piezoresistive coefficient has a value selected from the range of 0.01 to 100 Ω per μm of cantilever deflection. In another embodiment, the second piezoresistive coefficient has a very small value, preferably selected from the range of 0 to 1 Ω per μm of cantilever deflection. It is also preferred that the first and second piezoresistive sensors are positioned in the cantilever close to one another such that they have a substantially identical temperature, for example a temperature within 1 $^{\circ}\text{C}$, 5 $^{\circ}\text{C}$, 10 $^{\circ}\text{C}$, or 25 $^{\circ}\text{C}$ of one another. In some embodiments, the first and second piezoresistive sensors are positioned less than 10 μm , 25 μm , 50 μm , 100 μm , or 250 μm from one another. In an embodiment, the two piezoresistive strain sensors are positioned near the fixed end of a cantilever,

such that they experience a maximum strain when the cantilever is deflected. In an embodiment, the piezoresistive strain sensors are positioned at a distance from the fixed end less than 50% of the total length of the cantilever. In some embodiments, the first and second piezoresistive sensors are positioned less than 10 μm , 25 μm , 50 μm , 100 μm , or 250 μm from the fixed end of the cantilever.

[038] In a preferred embodiment, the microcantilever is comprised of single-crystal silicon or poly-crystalline silicon. In a preferred embodiment, the first piezoresistive sensor is aligned along a first crystal axis of the cantilever and the second piezoresistive sensor is aligned along a second crystal axis of the cantilever. It is also preferred that the first crystal axis is a $\langle 110 \rangle$ direction in silicon and the second crystal axis is a $\langle 100 \rangle$ direction in silicon. In an embodiment, the piezoresistive sensors comprise doped silicon, preferably silicon doped with an element that is soluble in silicon and that modifies the silicon electronic properties, such as boron or phosphorous. It is also preferred that the piezoresistive sensors have a dopant concentration selected from the range of 10^{14} to 10^{20} dopants per cubic centimeter.

[039] In a preferred embodiment, the first and second piezoresistive sensors are substantially identical; that is, they have substantially identical compositions and substantially identical dimensions. Substantially identical piezoresistive sensors help to ensure that any changes in the resistance of the piezoresistive sensors due to temperature will be substantially identical. It is preferred that the first and second piezoresistive sensors are identical.

[040] In an alternative embodiment, the microhotplate devices of the present invention may also comprise a resistance sensing circuit electrically connected to the first and second piezoresistive sensors. An exemplary resistance sensing circuit comprises a Wheatstone bridge circuit. Such a Wheatstone bridge circuit can be useful for compensating for changes in the resistances of the first and second piezoresistive sensors due to temperature

and for sensing changes in resistance due to surface stresses or cantilever deflections.

[041] In an embodiment, the heater-thermometer of the microhotplate devices of the present invention comprise doped silicon, preferably silicon doped with an element that is soluble in silicon and that modifies the silicon electronic properties, such as boron or phosphorous. It is also preferred that the heater-thermometer regions have a dopant concentration selected from the range of 10^{14} to 10^{20} dopants per cubic centimeter. In some embodiments the heater-thermometer is positioned either near the free end or near the fixed end of the cantilever. In other embodiments, the heater-thermometer comprises substantially an entire surface area of the cantilever. In exemplary embodiments, the heater-thermometer is partitioned among several regions of the cantilever. In other embodiments, the cantilever and/or heater-thermometer may have any shape, including shapes selected from the group consisting of square, rectangular, circular, U-shaped, ladder shaped, or any other shape into which the cantilever and/or heater-thermometer can be formed or patterned.

[042] The microhotplates devices of the present invention may also comprise other regions of doped silicon or regions of metal, useful as electrical traces or contacts or useful for delivering current to the heater-thermometer regions of the microhotplate. Use of highly doped silicon is preferred for some embodiments because it is capable of carrying high current densities, for example densities capable of producing temperatures in the heater-thermometer regions up to 1300 °C.

[043] In an exemplary embodiment, the first and second piezoresistive sensors are in thermal communication with the heater-thermometer of the cantilever. Having the piezoresistive sensors in thermal communication with the heater-thermometer ensures that the temperature of the piezoresistive sensors can be controlled by the heater-thermometer and that the piezoresistive sensors can have substantially identical temperatures.

[044] The invention may be further understood by the following non-limiting example.

EXAMPLE 1: Microcantilever hotplates with temperature-compensated piezoresistive strain sensors

[045] This example describes the design, fabrication, and characterization of microcantilever hotplates having both a resistive heater and temperature-compensated a piezoresistive strain gauge. The heater was defined near the cantilever free end and the piezoresistive strain gauges were integrated near the clamped base. To realize temperature compensation, a pair of identical piezoresistors was defined in close proximity. One piezoresistor was aligned to the $\langle 110 \rangle$ crystal direction where the piezoresistive coefficient is maximized and the other one was aligned to the $\langle 100 \rangle$ crystal direction where the piezoresistive coefficient was nearly zero. The fabricated devices exhibit excellent temperature compensation, with a 20 times reduction in temperature sensitivity. The deflection sensitivity shifted 10% for heating up to 200 °C and cantilever deflection on the order of 10 μm . This work enables cantilever sensors that can measure temperature-dependant phenomena.

Introduction

[046] Since the invention of the atomic force microscope (AFM), microcantilevers have become one of the most frequently used microelectromechanical systems (MEMS) devices with applications ranging from scanning probe microscopy to bio/chemical sensing. Microcantilevers are often functionalized by introducing current traces, piezoresistive or piezoelectric materials to realize specific applications. Heatable microcantilevers having either doped single-crystalline or polycrystalline silicon or patterned metal traces allow current flow so that they can be heated by means of Joule heating. This heating in microcantilevers can be used for bimorph actuation, thermomechanical data storage, thermal displacement sensing in contact and tapping modes, novel nano-material synthesis, nanoscale thermal analysis and nanoscale thermal manufacturing. Recently,

cantilever type micro-hotplates have been reported as an alternative platform for calorimetry. A cantilever type micro-hotplate fabricated based on porous silicon technology has been introduced and several microcantilever hotplates were designed, fabricated, and characterized to investigate response time and temperature uniformity.

[047] Microcantilevers with integrated piezoresistive strain sensors are mainly used to replace optical deflection sensing but are also employed in various sensing applications such as gas flow sensing, acceleration sensing, microjet measurements and bio/chemical sensing. Especially as bio/chemical sensors, piezoresistive microcantilevers are often prepared with a selective coating sensitive to a specific analyte. Analyte adsorption induces static deflection by creating a surface stress, and thus embedded piezoresistors can measure analyte adsorption.

[048] Microcantilevers having both resistive heaters and piezoresistors can offer simultaneous heating and sensing. These hybrid types have been used as multi-functional scanning probes in thermomechanical data storages or calorimetry and microbalance applications where the integrated piezoresistors were usually optimized for free-end deflection by a point load and not for surface stress loading. Similarly, microcantilevers with the ability of independent heating and sensing operation that have high sensitivity to surface stress could be used for a variety of sensor applications. One example would be calorimetry of a material adhered to the cantilever surface. Chemical processes such as melting and evaporation and chemical reactions between substances could be triggered by the heaters while the changes in the surface stresses on the cantilever are monitored and can give information about the material or reaction properties. Other examples include biochemical sensing, where one might wish to interrogate the temperature-dependence of biochemical binding to a microcantilever. Recent work has shown the usefulness of cantilever sensors for temperature-dependent biochemical sensing, although heating was global rather than local, and

deflection sensing was performed using far-field optics. Neither the heater nor the deflection sensor was fabricated into the cantilever itself.

[049] One strategy for suppressing unwanted signals, such as temperature drift, in piezoresistive cantilever sensors is to fabricate cantilever pairs for a differential measurement. Two microcantilevers with identical piezoresistive strain gauges can be arrayed closely and interfaced in a Wheatstone bridge to cancel temperature drift with the assumption they have the same temperature progression. However, this approach would not be appropriate for cases in which the two cantilevers experience different temperatures. This could be the case when a reactive coating modifies the thermal properties of one cantilever. Temperature deviations between the two devices can also be caused by the system environment, *e.g.* by thermal gradients due to gas flow directions. On-chip temperature compensation for piezoresistive cantilever sensors has been demonstrated, but these cantilevers did not have integrated heater-thermometers. Furthermore, all previous approaches to on-chip temperature compensation use the principle of a Wheatstone bridge circuit on the cantilever, a method which assumes unidirectional, equal stress in all resistors. However, for chemical sensing, in which a reactive layer causes a surface stress on the silicon surface, the stress distribution in the cantilever is complex and three-dimensional. Therefore, it is favorable to incorporate independent sensors for stress and temperature in the cantilever to correct the effect of thermal variations on the mechanical signal.

[050] This example describes the design, fabrication, and characterization of microcantilever hotplates with temperature-compensated piezoresistive strain gauges. Two different cantilever designs with the same surface area have integrated heaters along the cantilever edges and a pair of piezoresistors for temperature compensation near the clamped base. The fabricated devices showed successful integration of resistive heaters and piezoresistors and their electrical and thermal behaviors were thoroughly tested. Excellent temperature compensation of the deflection signal was confirmed, such that effect of the heater operation was negligible to the

piezoresistive readout. These microcantilever hotplates could enable simultaneous calorimetric and thermogravimetric measurements by operating the heater and the piezoresistor pair together.

Design and Fabrication

[051] Fig. 1 shows the two cantilever designs. The length and width of cantilever **A** are each 200 μm , while cantilever **B** is 300 μm long and 133 μm wide. The two cantilevers have approximately the same surface area. Each cantilever goes through two boron doping procedures, a high doping step to create low-resistance electrical traces, and a low doping step to create high resistance regions for strain and temperature sensing. The specifics of the doping steps are described later. The heater trace is created with the more highly doped silicon, and follows the edge of the cantilever. Both cantilever types have wide current traces on the cantilever edges in length direction and a narrow path at the free end, thus concentrating the majority of the resistance and therefore largest heat generation at the free end. The size of the narrow resistive heater at the free end is 130 $\mu\text{m} \times 5 \mu\text{m}$ for type **A** and 63 $\mu\text{m} \times 5 \mu\text{m}$ for type **B**. The doped silicon piezoresistor and the temperature-compensating resistor near the cantilever base are made up of the low-doped silicon. The size of each resistor is 20 $\mu\text{m} \times 10 \mu\text{m}$ as shown in Fig. 1. These elements are connected by highly-doped, lowly resistive electrical traces, so that the resistance of the traces is dominated by the piezoresistors and not the electrical connection.

[052] The temperature compensation is accomplished through two highly resistive elements, one of which being sensitive to both strain and temperature, and the other one being sensitive to temperature but relatively insensitive to strain. The strain-sensing resistive element is the high resistance element on the centerline of the cantilever, such that the current flow direction in the piezoresistor is aligned completely in the highly sensitive $\langle 110 \rangle$ crystal direction of the silicon device layer. The second resistor of equal dimensions is placed in close proximity to the first piezoresistor, but with

an angle of 45° to it, therefore aligning it to the $\langle 100 \rangle$ crystal direction. The piezoresistive coefficients for p-type silicon in this direction are zero, so that the $\langle 100 \rangle$ resistor should be insensitive to stresses in the cantilever, whereas the sensitivity of the $\langle 110 \rangle$ resistor is maximized. A similar concept has been demonstrated previously for an AFM cantilever with resistors in its two legs, which are angled at 45° to each other and provide effective temperature compensation. To cancel the effect of temperature changes, the two resistors can be connected in one branch of a Wheatstone bridge and supplemented by two additional resistors. For chemical sensing, the other branch of the Wheatstone bridge can be formed by the resistors in a second cantilever on the same chip, thus cancelling effects that alter the surface stress other than those caused by the exposure to the analyte.

[053] Fig. 2 shows the six major fabrication steps to make the microcantilever hotplates with temperature-compensated piezoresistors. The fabrication process started with an n-type silicon-on-insulator (SOI) wafer of orientation $\langle 100 \rangle$, where the silicon device layer was $2\ \mu\text{m}$, the buried oxide (BOX) layer was $1\ \mu\text{m}$, and the silicon handle layer was $400\ \mu\text{m}$. To precisely control the cantilever thickness, silicon dioxide was thermally grown on the wafer surface and etched away with high selectivity. This process, which slowly but evenly consumes the silicon of the device layer, was repeated several times while the remaining device layer thickness was monitored and the growth parameters were adjusted accordingly. After the thinning of the device layer to meet the target thickness of $1\ \mu\text{m}$, the beam structures were patterned with photoresist (Shipley 1827) and etched into the silicon device layer using a Bosch process in an inductively coupled plasma (ICP) etcher.

[054] Two implantation steps were performed with hard-baked positive photoresist (Shipley 1827) as a mask for ion implantation. The first doping step created current traces and heaters by ion implantation with the parameters shown in Table 1. Then, the photoresist implantation mask was removed and a $200\ \text{nm}$ thick layer of silicon dioxide was deposited using plasma enhanced chemical vapor deposition (PECVD) to prevent the dopants

from diffusing from silicon to ambient during the subsequent heat treatment. The heat treatment was performed in a furnace with nitrogen atmosphere and is used to anneal the silicon and to achieve a more uniform dopant distribution as shown in Fig. 3. After the heat treatment, the previously deposited silicon dioxide was removed in buffered oxide etch (BOE) to reveal the silicon for the second implantation step. The second doping step was performed to make the piezoresistors by ion implantation with the low doping parameters in Table 1. Similar to the first doping step, a 200 nm thick silicon dioxide was subsequently deposited and the processed wafers were rapid-thermal annealed to recrystallize the silicon structure damaged during ion implantation. The vias connecting the doped silicon to the metal layer were defined with the ICP etcher and aluminum metallization was performed using electron beam deposition in combination with a lift off process. Afterwards, a 30-minute sintering step at 400 °C in a forming gas was done to allow inter-diffusion of doped silicon and aluminum.

Table 1. Implantation and heat treatment parameters and expected sheet resistances from doping simulations for high and low-doped areas.

	High-doped areas	Low-doped areas
Implantation dose (cm ⁻²)	3×10 ¹⁵	2×10 ¹³
Implantation energy (keV)	120	20
Anneal temperature (°C)	1000	1000
Anneal time (min)	60	30
Expected sheet resistance (Ω/□)	32.85	2182

[055] For the final release, the handle layer was etched from the backside using the Bosch process in the ICP etcher and the BOX layer was removed in 49% hydrofluoric acid (HF). To circumvent the problem that HF attacked the aluminum, an additional photolithography step was added after the backside

through-wafer etch. The same lithography mask that revealed the trenches on the backside of the wafer to get free-hanging cantilevers was used again to cover the same geometry on the topside leaving only the cantilever and the surrounding trench area uncovered during the BOX layer etching. This additional step effectively protected the aluminum from the HF and resulted in significantly increased yield. The final yield was better than 90% across the 4-inch wafer. Fig. 3 shows scanning electron micrographs of the released devices.

Simulation

[056] Since the shape and placement of the resistors are fixed by the preceding considerations, the electrical connections between the resistors and to the bond pads are made with highly boron-doped silicon. There are several advantages to using high-doped silicon instead of using the same doping level as for the resistors or using a different electrically conductive material. The resistance of the current traces made of high-doped silicon will be small compared to the resistance of the piezoresistors, which ensures a high ratio of resistance change to initial resistance during sensing operation, and thus a good sensitivity. The high-doped silicon also has a reduced piezoresistive coefficient, so that unwanted resistance change in the conductor areas is small. Furthermore, doping the silicon will have very little effect on the stress state inside the cantilever, whereas deposited layers might cause intrinsic stresses and lead to initial device deformation. Finally, the coefficients of thermal expansion for intrinsic and doped silicon are expected to be very similar, so that changes in the cantilever stress state due to heating are minimized. The last requirement is impossible to achieve with most other electrically conductive materials that could otherwise be used to shape the current paths. The described arrangement of the piezoresistor and the additional resistor is used for both cantilever types.

[057] A design requirement for the piezoresistor, the additional resistor and the heater is that the total resistance for each element is below 5 k Ω . This

resistance is sufficiently low to be driven with conventional electronics and to reduce noise, but is high compared to other resistances in the system. Since the two piezoresistors are twice as long as they are wide, the sheet resistance of the low-doped silicon must be below $2.5 \text{ k}\Omega/\square$. The high doping step must produce electrical connections to the resistors and are small compared to the piezoresistor resistance. The integrated heaters are about 40-50 times longer than they are wide, and so the heaters must be formed by the high doping step.

[058] Fig. 4 shows the SSUPREME3 simulation results using implantation and heat treatment parameters from Table 1. It can be seen that the low-doped areas have a sheet resistance almost two orders of magnitude higher than that of the high-doped areas. From the expected concentration distribution of the boron atoms, the junction depth of the piezoresistor is about one third of the cantilever thickness. For the high-doped areas that form the connecting traces of the piezoresistor and the additional resistor as well as the heater structures, the dopants are more evenly distributed which acts to reduce current density during heating.

Characterization

[059] Electrical characterization. The basic electrical testing was performed following characterization techniques described in [J. Lee, T. Beechem, T. L. Wright, B. A. Nelson, S. Graham, and W. P. King, J. Microelectromech. Syst., 15 (2006) 1644-1655]. Fig. 5 shows the circuit for electrical testing, where the doped resistors (heater, $\langle 100 \rangle$ piezoresistor, or $\langle 110 \rangle$ piezoresistor) were configured in series with a precision power resistor. The series power resistor can be referred to as the “sense” resistor in the 2-resistor bridge design. Fig. 6(a) shows heater resistances as a function of the applied voltage to the heater (heater voltage). The measured resistances are nearly constant at low voltages since the Joule heating does not increase the device temperature significantly. As the heater voltage is increased, the heater resistance increases due to the positive temperature coefficient of resistance (TCR) at

lower temperatures. Once the heater voltage reaches a critical point, the heater resistance drops suddenly. This is the well-known thermal runaway of doped silicon. The sheet resistance of the high-doped areas calculated from the heater resistance and the geometry is between 29 and 32 $\Omega/\square$, which is very close to the predicted value of 32.85 $\Omega/\square$.

[060] Fig. 6(b) shows the dissipated power in the heater as a function of the heater voltage. There is a strong increase in power near the thermal runaway as the resistance drops and thus the current increases. The type **A** cantilever can dissipate more power than the type **B** cantilever before thermal runaway occurs. This can be attributed to the relatively closer placement of the heater to the silicon handle, which acts as a heat sink.

[061] The same electrical characterization was performed for the piezoresistors in $\langle 110 \rangle$ direction and $\langle 100 \rangle$ direction, shown in Figs. 6(c) and (d). The room temperature resistance is very similar for both types and directions, which is expected since the resistor geometries are the same. The alignment of the crystal directions does not affect the resistance, which is important in order to achieve a well-balanced bridge circuit for temperature compensation. Compared to the small surface area of the resistors, the dissipated power in the piezoresistors at the thermal runaway point is very large, which is due to the proximity of the resistors to the clamped base.

[062] The relationship between resistance and voltage in Figs. 6(a) and (c) and hence also the information about power vs. voltage in Figs. 6(c) and (d) were obtained after the device was powered past the thermal runaway point several times. These thermal cycles were found to reduce the room temperature resistance and also modify the behavior of the resistance at increased voltages. This burn-in period for heated cantilevers has been previously investigated.

[063] The electrical characteristics of two cantilevers on one chip were within 0.5 % from each other, so that only results for one cantilever are shown

here. Their similar behaviors make them well-suited for differential operation to cancel out any parasitic effects.

[064] Temperature characterization. Raman spectroscopy was used to determine the local temperature in the center of the heater trace at the free end. Fig. 7 shows the hot spot temperature of each device type for five different power levels. The long type **B** devices reach the same temperatures as type **A** devices at lower cantilever powers. After the relationship between power and hot spot temperature was determined for each cantilever type the same devices were used to investigate the spatial distribution of the temperature. For this purpose, the power was fixed at a level for which a corresponding hot spot temperature of 200 °C was expected and Raman measurements were taken in eleven different locations along the cantilever length direction between the hot spot and the clamped edge. The temperature distribution is plotted vs. the relative position, *i.e.* the ratio between distance from the free end and cantilever length, in Fig. 8(a). This plot style was chosen because it allows for a better comparison of the temperature trends of devices with different lengths. Although the distributions are almost linear and very close to each other, there are differences between the two heater shapes. Fig. 8(b), which shows the temperature distribution along the width direction, seems to confirm this trend although the differences are marginal. The data points at about 2.5 and 82.5% in Fig. 8(a) and at 45% in Fig. 8(b) show somewhat higher temperatures than expected from the adjacent data points. Since these positions were in the high-doped silicon areas, the change in the temperature slope could have been caused by the lower thermal conductivity. The deviation could also be caused by the measurement error of the Raman system because the relationship between shift in peak position and temperature is calibrated for the intrinsic silicon. The accuracy of the temperature measurements in the intrinsic silicon areas is within 5 K over the tested temperature range.

[065] Sensitivity characterization. The two cantilevers, whose electrical and thermal properties were characterized in the previous sections, were used to perform deflection sensitivity measurements with the same setup as described in [B. W. Chui, L. Aeschimann, T. Akiyama, U. Staufer, N. F. de Rooij, J. Lee, F. Goericke, W. P. King, and P. Vettiger, Rev. Sci. Instrum., 78 (2007) 043706]. One of the main objectives of these investigations is to quantify the ability to perform temperature compensated deflection measurements using the previously discussed temperature compensation scheme. For this purpose, the characteristics of either the $\langle 110 \rangle$ piezoresistor, the $\langle 100 \rangle$ piezoresistor, or both piezoresistors were tested during cantilever deflection. These resistors were configured in two Wheatstone bridge circuits, shown in Fig. 9. Fig. 10(a) shows the bridge output as a function of tip deflection for the type **B** cantilever. As expected, the $\langle 100 \rangle$ piezoresistor is very insensitive to cantilever deflection, while the $\langle 110 \rangle$ resistor is quite sensitive to cantilever deflection. When both piezoresistors are included in the Wheatstone bridge, the resulting signal is nearly identical to the signal from the $\langle 110 \rangle$ resistor alone.

[066] The three experimental runs were repeated, but instead of deflecting the free end of the type B cantilever, the hot spot temperature was modulated by powering the integrated heater trace. Fig. 10(b) shows the bridge output for the heated, undeflected cantilever. For a hot spot temperature of 100 °C, the circuit output for the $\langle 100 \rangle$ resistor is considerably large. Without temperature compensation, the signal from this piezoresistor is equivalent to a deflection of more than 100 μm . When connected alone, the bridge output signals of the $\langle 100 \rangle$ and the $\langle 110 \rangle$ resistors are very similar, indicating that their temperature sensitivities are also very similar. When both resistors are connected in the Wheatstone bridge, the output is much smaller because the resistance changes due to the temperature variation cancel. The temperature sensitivity of the temperature-compensation piezoresistor pair is reduced by a factor of 20 compared to the uncompensated piezoresistor configuration at a hot spot temperature of 100 °C.

[067] The small signal that remains is assumed to be caused by the difference in the two resistors' average temperature due to their different locations on the cantilever. While only the results from type **B** are shown, type **A** cantilever showed similar trends for both tip deflection and temperature modulation. However, the type **A** cantilever shows higher deflection sensitivity because an equivalent absolute change in cantilever deflection will cause greater stresses in these square-shaped devices than in the longer and narrower type B device. From Fig. 10, it can be concluded that the novel resistor arrangement on the cantilever can indeed be used to measure changes in the cantilever stress with greatly reduced parasitic signals from temperature changes and without sacrificing mechanical sensitivity.

[068] While it is important for the cantilever to be sensitive to deflections with low sensitivity to temperature changes, it is even more important for the cantilever to have consistent deflection sensitivity over a large temperature range. This may be thought of as the temperature coefficient of tip deflection sensitivity (TCS), which must be minimized. Fig. 11(a) shows the temperature-compensated bridge output signal as a function of tip deflection for the type **B** cantilever. The cantilever was deflected for six different hot spot temperatures under self-heating. The deflection sensitivity is not a very strong function of the hot spot temperature in the range 25-200 °C. Fig. 11(b) shows the normalized deflection sensitivities vs. temperature derived from the slope of linear fits to the data points in Fig. 11(a). The slope of the linear fits in the resulting figure represents the TCS and it is on the order of $-5 \times 10^{-4} \text{ K}^{-1}$. Overall, the deflection sensitivity of the temperature-compensated cantilevers under self-heating varies by about 10% over 200 °C. Both cantilever types have very similar values and their difference is not significant compared to the scattering of the data points.

[069] Table 2 summarizes typical characterization results for both cantilever types. Type **A** has smaller hot spot temperature sensitivity, which indicates that is well-suited for static chemical sensing applications which accompany temperature. However, for oscillatory operation it might be favorable to use

the type **B** cantilever, as the longer cantilever might be more sensitive to adherent mass. The lower hot spot temperature sensitivity of the type **A** cantilever can be explained with the distance from the piezoresistor pair to the current traces. Due to the short and wide geometry, the type **A** cantilever has better temperature uniformity specifically for width direction upon the integrated heater operation. The temperature uniformity will likely improve with an optimized heater design.

Table 2. Summary of properties for both cantilever types.

	A	B
Room temperature resistance of heater (k Ω)	1.49	1.44
Room temperature resistance of <110> resistor (k Ω)	3.17	3.56
Room temperature resistance of <100> resistor (k Ω)	3.56	3.78
Heater voltage at thermal runaway (V)	10.8	8.1
Heater resistance at thermal runaway (k Ω)	2.27	2.06
Heater power at thermal runaway (mW)	51.4	31.8
Base temperature at 200 °C hot spot temperature (°C)	46.2	42.9
Room temperature tip deflection sensitivity (mV/V- μ m)	0.268	0.0931
Hot spot temperature coefficient of tip deflection sensitivity (K ⁻¹)	-5.97×10^{-4}	-5.86×10^{-4}
Hot spot temperature sensitivity (μ V/V-K)	-4.84	-8.74

Conclusions

[070] Silicon microcantilevers with integrated heater and temperature compensation structures were designed, fabricated, and characterized. Two different cantilever geometries were compared. The hot spot temperatures that can be reached with these devices are greater than 500 °C without

thermal failure. The temperature was found to decrease about linearly along the cantilever with the hot spot temperature at the free end and the coldest point at the clamped substrate, while the temperature distribution in the width direction is very uniform. Resistor structures with different crystal directions but in close proximity on the cantilever surface were also implemented in the design and their properties were characterized. The experiments showed that those resistors aligned in $\langle 110 \rangle$ direction showed high sensitivity to both cantilever deflection and temperature changes, whereas the resistors in $\langle 100 \rangle$ direction were only sensitive to temperature changes and had very little deflection sensitivity. When combined in a Wheatstone bridge circuit, these resistors can greatly reduce temperature-induced error without sacrificing deflection sensitivity. Under temperature compensation, the deflection sensitivity is not a strong function of the temperature, enabling the accurate sensing of deflections during heater operation or exposure to external heat sources.

Figure Descriptions

[071] Figure 1 shows a schematic of two types of microcantilever hotplates with temperature-compensated piezoresistors. Their sizes are $200\text{ }\mu\text{m} \times 200\text{ }\mu\text{m}$ (type **A**) and $300\text{ }\mu\text{m} \times 133\text{ }\mu\text{m}$ (type **B**), respectively. Thus, both types have approximately the same surface area. There are a resistive heater near the free end and two piezoresistors near the clamped base. The zoom-in shows a piezoresistor parallel to $\langle 110 \rangle$ direction and a piezoresistor parallel to $\langle 100 \rangle$ direction.

[072] Figure 2 shows a schematic of six major fabrication processes to make the microcantilever hotplates with temperature-compensated piezoresistors. (a) beam and metal pad outlines are etched into the device layer (b) heater and high doped regions are boron implanted (c) piezoresistors are boron implanted (d) vias are made and aluminum metallization is followed (e) silicon handle layer is etched from the backside using ICP (f) the buried oxide layer is etched in HF.

[073] Figure 3 shows scanning electron micrographs of the fabricated microcantilever hotplates.

[074] Figure 4 shows simulated boron concentration using SSUPREME3 for low and high doping processes. Solid and dashed lines are boron profiles before and after the heat treatment, respectively. From this, the junction depth of the low-doped piezoresistor is about one third of the cantilever thickness. Dopants are more evenly distributed for high-doped areas which acts to reduce current density upon heating.

[075] Figure 5 shows a testing circuit for electrical characterization. Either doped heater or piezoresistors are connected to a sense resistor (precision power resistor) which senses current and protect the device at high powers. V_{total} represents actual applied voltage to each doped resistor and V_{sense} represents voltage drop across the sense resistor.

[076] Figure 6 shows data illustrating (a) Heater resistance and (b) heater power vs. applied voltage to the heater – heater voltage – for two cantilever types (c) Piezoresistor resistance and (d) piezoresistor power vs. applied voltage to the piezoresistors – piezoresistor voltage – in $\langle 110 \rangle$ and $\langle 100 \rangle$ direction for two cantilever types.

[077] Figure 7 shows data illustrating the hot spot temperature of each device type for five different power levels from laser Raman thermometry. The long type **B** devices reach the same temperatures as type **A** devices at lower cantilever powers.

[078] Figure 8 shows measurements of local temperature vs. relative position for type **A** and **B** (a) along the cantilever length, where 0% is the center of the free end and 100% is the center of the clamped base and (b) along the cantilever width, where 0% is at the cantilever center in length and width direction and 50% is at the edge in width direction and in the center of the length direction.

[079] Figure 9 shows two circuit configurations using the on-chip resistors in the Wheatstone bridge. (a) To test the individual elements alone, the resistor in either the $\langle 110 \rangle$ or the $\langle 100 \rangle$ crystal direction was used. (b) To test the compensation characteristics of the resistors, both $\langle 110 \rangle$ and $\langle 100 \rangle$ crystal direction piezoresistors were used.

[080] Figure 10 shows measured characteristics of the various resistors from a type **B** cantilever. The bridge output signal is shown for the $\langle 110 \rangle$ resistor or the $\langle 100 \rangle$ resistor using the circuit in Fig. 9(a), and for both resistors when in the circuit of Fig. 9(b). The bridge output signal is shown for (a) deflection of the unheated cantilever and (b) heating of the cantilever with no deflection. The uncompensated cantilever is sensitive to both temperature and deflection, while the compensated cantilever is sensitive to deflection but relatively insensitive to temperature.

[081] Figure 11 shows a summary of cantilever deflection sensitivity up to 200 °C. (a) Bridge output signal with both piezoresistors configured vs. tip deflection of the type B cantilever for six different hot spot temperatures (b) Normalized tip deflection sensitivity vs. hot spot temperature for both cantilever types. The cantilever deflection sensitivity changes by about 10% over the temperature range measured, which is a factor of 20 improvement over the uncompensated case.

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[0123] U.S. Patent Application Publication No. US2006/0207317.

STATEMENTS REGARDING INCORPORATION BY REFERENCE AND VARIATIONS

[0124] All references throughout this application, for example patent documents including issued or granted patents or equivalents; patent application publications; and non-patent literature documents or other source

material; are hereby incorporated by reference herein in their entireties, as though individually incorporated by reference, to the extent each reference is at least partially not inconsistent with the disclosure in this application (for example, a reference that is partially inconsistent is incorporated by reference except for the partially inconsistent portion of the reference).

[0125] All patents and publications mentioned in the specification are indicative of the levels of skill of those skilled in the art to which the invention pertains. References cited herein are incorporated by reference herein in their entirety to indicate the state of the art, in some cases as of their filing date, and it is intended that this information can be employed herein, if needed, to exclude (for example, to disclaim) specific embodiments that are in the prior art. For example, when a compound is claimed, it should be understood that compounds known in the prior art, including certain compounds disclosed in the references disclosed herein (particularly in referenced patent documents), are not intended to be included in the claim.

[0126] When a group of substituents is disclosed herein, it is understood that all individual members of those groups and all subgroups, including any isomers and enantiomers of the group members, and classes of compounds that can be formed using the substituents are disclosed separately. When a compound is claimed, it should be understood that compounds known in the art including the compounds disclosed in the references disclosed herein are not intended to be included. When a Markush group or other grouping is used herein, all individual members of the group and all combinations and subcombinations possible of the group are intended to be individually included in the disclosure.

[0127] Every formulation or combination of components described or exemplified can be used to practice the invention, unless otherwise stated. Specific names of compounds are intended to be exemplary, as it is known that one of ordinary skill in the art can name the same compounds differently. When a compound is described herein such that a particular isomer or

enantiomer of the compound is not specified, for example, in a formula or in a chemical name, that description is intended to include each isomers and enantiomer of the compound described individual or in any combination. One of ordinary skill in the art will appreciate that methods, device elements, starting materials, and synthetic methods other than those specifically exemplified can be employed in the practice of the invention without resort to undue experimentation. All art-known functional equivalents, of any such methods, device elements, starting materials, and synthetic methods are intended to be included in this invention. Whenever a range is given in the specification, for example, a temperature range, a time range, or a composition range, all intermediate ranges and subranges, as well as all individual values included in the ranges given are intended to be included in the disclosure.

[0128] As used herein, “comprising” is synonymous with "including," "containing," "having," or "characterized by," and is inclusive or open-ended and does not exclude additional, unrecited elements or method steps. As used herein, "consisting of" excludes any element, step, or ingredient not specified in the claim element. As used herein, "consisting essentially of" does not exclude materials or steps that do not materially affect the basic and novel characteristics of the claim. Any recitation herein of the term “comprising”, particularly in a description of components of a composition or in a description of elements of a device, is understood to encompass those compositions and methods consisting essentially of and consisting of the recited components or elements. The invention illustratively described herein suitably may be practiced in the absence of any element or elements, limitation or limitations which is not specifically disclosed herein.

[0129] The terms and expressions which have been employed are used as terms of description and not of limitation, and there is no intention in the use of such terms and expressions of excluding any equivalents of the features shown and described or portions thereof, but it is recognized that various modifications are possible within the scope of the invention claimed. Thus, it

should be understood that although the present invention has been specifically disclosed by preferred embodiments and optional features, modification and variation of the concepts herein disclosed may be resorted to by those skilled in the art, and that such modifications and variations are considered to be within the scope of this invention as defined by the appended claims.

CLAIMS

We claim:

1. A microhotplate comprising:
 - a. a cantilever having a fixed end and a free end;
 - b. a pair of piezoresistive sensors integrated into said cantilever near said fixed end, wherein the first piezoresistive sensor is aligned along a first crystal axis of said cantilever and has a first piezoresistive coefficient, and wherein the second piezoresistive sensor is aligned along a second crystal axis of said cantilever and has a second piezoresistive coefficient that is less than said first piezoresistive coefficient; and
 - c. a heater-thermometer integrated into said cantilever.
2. The microhotplate of claim 1 wherein said second piezoresistive coefficient has a value selected from the range of 0 to 1 Ω per μm of cantilever deflection.
3. The microhotplate of claim 1 wherein said first piezoresistive coefficient has a value selected from the range of 0.1 to 100 Ω per μm of cantilever deflection.
4. The microhotplate of claim 1 wherein the distance between said fixed end of said cantilever and said piezoresistive sensors is less than 50% of the total length of said cantilever.
5. The microhotplate of claim 1 wherein said first and second piezoresistive sensors are positioned in said cantilever in proximity to each other such that they have a substantially identical temperature.
6. The microhotplate of claim 1 wherein said cantilever comprises single crystal silicon.

7. The microhotplate of claim 6 wherein said first crystal axis is a <110> direction in silicon and said second crystal axis is a <100> direction in silicon.
8. The microhotplate of claim 1 wherein said first and second piezoresistive sensors comprise doped silicon.
9. The microhotplate of claim 8 wherein said piezoresistive sensors are doped with an element selected from the group consisting of phosphorous, boron, or other elements that are soluble in the material of the cantilever and that change the cantilever material properties.
10. The microhotplate of claim 8 wherein said piezoresistive sensors have a dopant concentration selected from the range of 10^{14} to 10^{20} dopants per cubic centimeter.
11. The microhotplate of claim 1 wherein said first and second piezoresistive sensors have substantially identical dimensions and substantially identical compositions.
12. The microhotplate of claim 1 further comprising a resistance sensing circuit electrically connected to said first and second piezoresistive sensors.
13. The microhotplate of claim 12 wherein said resistance sensing circuit comprises a Wheatstone bridge circuit.
14. The microhotplate of claim 12 wherein said Wheatstone bridge circuit compensates for a change in the resistance of said first and second piezoresistive sensors due to temperature.
15. The microhotplate of claim 1 wherein said heater-thermometer comprises a resistive heater.

16. The microhotplate of claim 1 wherein said heater-thermometer comprises doped silicon.
17. The microhotplate of claim 16 wherein said heater-thermometer is doped with an element selected from the group consisting of phosphorous, boron, or other elements that are soluble in the material of the cantilever and that change the cantilever material properties.
18. The microhotplate of claim 16 wherein said heater-thermometer has a dopant concentration selected from the range of 10^{14} to 10^{20} dopants per cubic centimeter.
19. The microhotplate of claim 1 wherein said heater-thermometer is positioned at said free end of said cantilever.
20. The microhotplate of claim 1 wherein said heater-thermometer comprises substantially an entire surface area of said cantilever.
21. The microhotplate of claim 1 wherein said heater-thermometer is capable of producing temperatures up to 1300 °C in said cantilever.
22. The microhotplate of claim 1 wherein said first and second piezoresistive sensors are in thermal communication with said heater-thermometer.
23. A method of sensing a surface stress of a microhotplate, the method comprising:
 - a. providing a microhotplate, said microhotplate comprising:
 - i. a cantilever having a fixed end and a free end;
 - ii. a pair of piezoresistive sensors integrated into said cantilever near said fixed end, wherein the first piezoresistive sensor is aligned along a first crystal axis of

said cantilever and has a first piezoresistive coefficient, and wherein the second piezoresistive sensor is aligned along a second crystal axis of said cantilever and has a second piezoresistive coefficient that is less than said first piezoresistive coefficient; and

- iii. a heater-thermometer integrated into said cantilever;
 - b. electrically connecting said first and second piezoresistive sensors in a Wheatstone bridge circuit; and
 - c. sensing a change in a resistance of said first piezoresistive sensor, thereby sensing a surface stress of said microhotplate.
24. The method of claim 23 wherein said Wheatstone bridge circuit compensates for a change in a resistance of said first and second piezoresistive sensors due to temperature.
25. The method of claim 23 wherein said first and second piezoresistive sensors are positioned in said cantilever in proximity to each other such that they have a substantially identical temperature.
26. A method of sensing a property of a compound with a microhotplate, the method comprising:
- a. providing a microhotplate, said microhotplate comprising:
 - i. a cantilever having a fixed end and a free end;
 - ii. a pair of piezoresistive sensors integrated into said cantilever near said fixed end, wherein the first piezoresistive sensor is aligned along a first crystal axis of said cantilever and has a first piezoresistive coefficient, and wherein the second piezoresistive sensor is aligned along a second crystal axis of said cantilever and has a

second piezoresistive coefficient less than said first piezoresistive coefficient; and

- iii. a heater-thermometer integrated into said cantilever;
 - b. contacting said free end of said cantilever with a compound;
 - c. providing a voltage or current to said heater-thermometer, thereby changing a temperature of said cantilever and compound and wherein said temperature change of said compound produces a surface stress in said cantilever; and
 - d. sensing a change in a resistance of said first piezoresistive sensor, thereby sensing a property of said compound.
27. The method of claim 26 wherein said property of said compound is selected from the group consisting of: a melting point of said compound, a boiling point of said compound, a binding energy of said compound, a heat capacity of said compound, a mechanical expansion of said compound, or a mechanical contraction of said compound.
28. The method of claim 26 wherein said surface stress is produced by an event selected from the group consisting of: a change in the state of said compound, evaporation of said compound, a change in the amount of said compound contacting said cantilever, a change in the extent of binding of said compound to said cantilever, a mechanical expansion of said compound, or a mechanical contraction of said compound.
29. The method of claim 26 further comprising sensing a resistance of said heater-thermometer, thereby sensing a temperature of said cantilever and said compound.
30. The method of claim 26 wherein said change in a resistance of said first piezoresistive sensor is sensed by a method comprising

electrically connecting said first and second piezoresistive sensors in a Wheatstone bridge circuit in a manner to compensate for a change in a resistance of said first and second piezoresistive sensors due to temperature.

31. A method of sensing a species bound to a microhotplate, the method comprising:
 - a. providing a first microhotplate, said microhotplate comprising:
 - i. a cantilever having a fixed end and a free end;
 - ii. a pair of piezoresistive sensors integrated into said cantilever near said fixed end, wherein the first piezoresistive sensor is aligned along a first crystal axis of said cantilever and has a first piezoresistive coefficient, and wherein the second piezoresistive sensor is aligned along a second crystal axis of said cantilever and has a second piezoresistive coefficient that is less than said first piezoresistive coefficient; and
 - iii. a heater-thermometer integrated into said cantilever;
 - b. providing a species capable of binding to said free end of said cantilever, wherein binding of said species to said cantilever produces a surface stress in said cantilever; and
 - c. sensing a change in a resistance of said first piezoresistive sensor, thereby sensing said species bound to said microhotplate.
32. The method of claim 31 wherein said free end of said cantilever comprises a substance capable of binding said species.

33. The method of claim 31 further comprising providing a voltage or current to said heater-thermometer, thereby changing the temperature of said cantilever.
34. The method of claim 31 wherein said resistance of said first piezoresistive sensor is sensed by a method comprising electrically connecting said first and second piezoresistive sensors in a Wheatstone bridge circuit.
35. The method of claim 34 wherein said Wheatstone bridge circuit compensates for a change in a resistance of said first and second piezoresistive sensors due to temperature.
36. The method of claim 31 further comprising:
 - a. providing a second microhotplate substantially identical to said first microhotplate, said second microhotplate comprising:
 - i. a cantilever having a fixed end and a free end;
 - ii. a pair of piezoresistive sensors integrated into said cantilever near said fixed end, wherein a first piezoresistive sensor is aligned along a first crystal axis of said cantilever and has a first piezoresistive coefficient, and wherein the second piezoresistive sensor is aligned along a second crystal axis of said cantilever and has a second piezoresistive coefficient that is less than said first piezoresistive coefficient; and
 - iii. a heater-thermometer integrated into said cantilever;
 - b. providing said second microhotplate in an environment substantially identical to said first microhotplate, wherein said environment does not have said species present; and

- c. electrically connecting said first and second piezoresistive sensors of said first microhotplate and said first and second piezoresistive sensors of said second microhotplate in a Wheatstone bridge circuit in a manner that compensates for changes in resistance of said first and second piezoresistive sensors of said first microhotplate due to effects other than said species binding to said free end of said cantilever of said first microhotplate.
- 37. A method of performing a temperature compensated deflection measurement, the method comprising:
 - a. providing a microhotplate, said microhotplate comprising:
 - i. a cantilever having a fixed end and a free end;
 - ii. a pair of piezoresistive sensors integrated into said cantilever near said fixed end, wherein the first piezoresistive sensor is aligned along a first crystal axis of said cantilever and has a first piezoresistive coefficient, and wherein the second piezoresistive sensor is aligned along a second crystal axis of said cantilever has a second piezoresistive coefficient that is less than said first piezoresistive coefficient; and
 - iii. a heater-thermometer integrated into said cantilever;
 - b. electrically connecting said first and second piezoresistive sensors in a Wheatstone bridge circuit in a manner to compensate for a change in a resistance of said first and second piezoresistive sensors due to temperature; and
 - c. sensing a change in a resistance of said first piezoresistive sensor due to a deflection of said cantilever, thereby

performing a temperature compensated deflection measurement.

38. The method of claim 37 further comprising providing a current or voltage to said heater-thermometer, thereby changing the temperature of said cantilever.

ABSTRACT

Microcantilever Heater-Thermometer with Integrated Temperature-Compensated Strain Sensor

The present invention provides microcantilever hotplate devices which incorporate temperature compensating strain sensors. The microcantilever hotplate devices of the present invention comprise microcantilevers having temperature compensating strain sensors and resistive heaters. The present invention also provides methods for using a microcantilever hotplate for temperature compensated surface stress measurements, chemical/biochemical sensing, measuring various properties of compounds adhered to the microcantilever hotplate surface, or for temperature compensated deflection measurements.

Figure 1