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(54) TEMPERATURE-DEPENDENT NANOSCALE CONTACT POTENTIAL MEASUREMENT TECHNIQUE AND DEVICE

(75) Inventor: William P. King, Champaign, IL (US)

Assignee: The Board of Trustees of the

University of Illinois, Urbana, IL (US)

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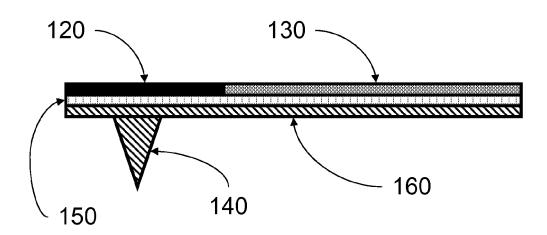
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Primary Examiner — Nicole Ippolito (74) Attorney, Agent, or Firm — Lathrop & Gage LLP

(57)ABSTRACT

The present invention provides a microcantilever capable of independently measuring and/or controlling the electrical potential and/or temperature of a surface with nanometer scale position resolution. The present invention also provides methods of manipulating, imaging, and/or mapping a surface or the properties of a surface with a microcantilever. The microcantilevers of the present invention are also capable of independently measuring and/or controlling the electrical potential and/or temperature of a gas or liquid. The devices and methods of the present invention are useful for applications including gas, liquid, and surface sensing, micro- and nano-fabrication, imaging and mapping of surface contours or surface properties.

38 Claims, 9 Drawing Sheets



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International Search Report and Written Opinion dated Oct. 4, 2012, for International Application No. PCT/US12/48326.

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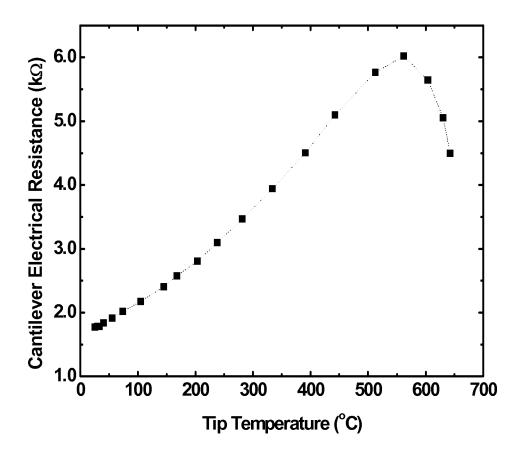
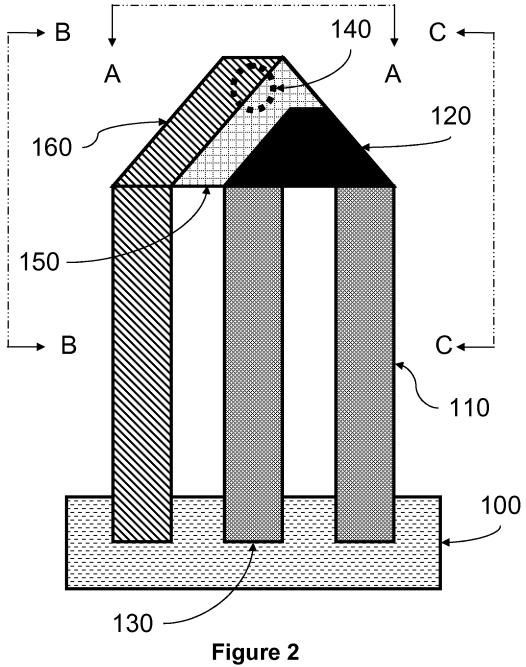


Figure 1



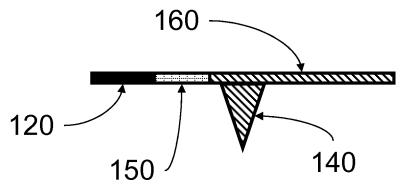


Figure 3

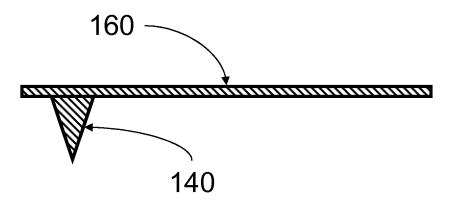


Figure 4

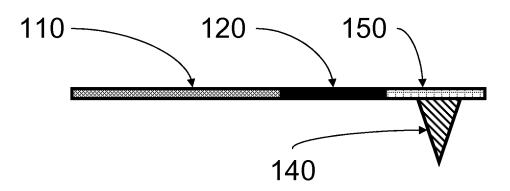
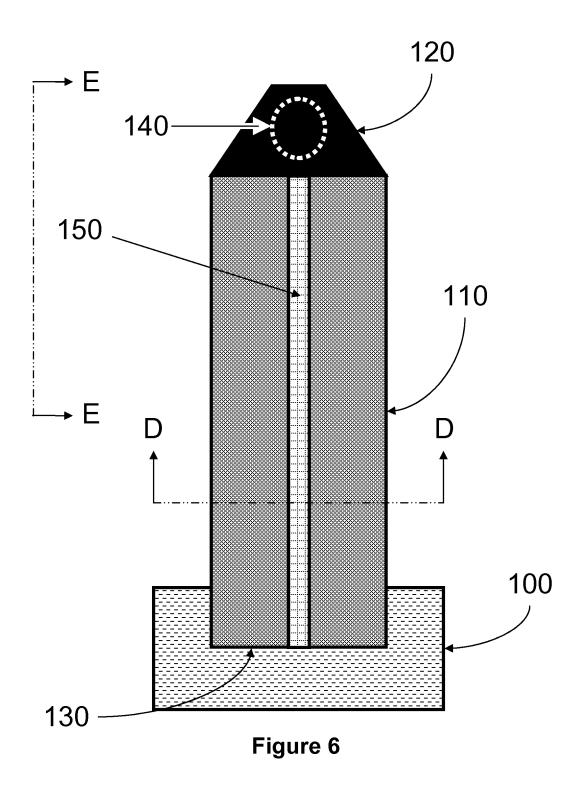


Figure 5



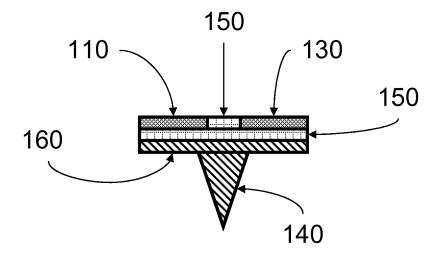


Figure 7

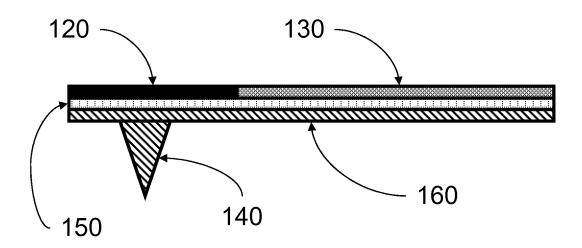
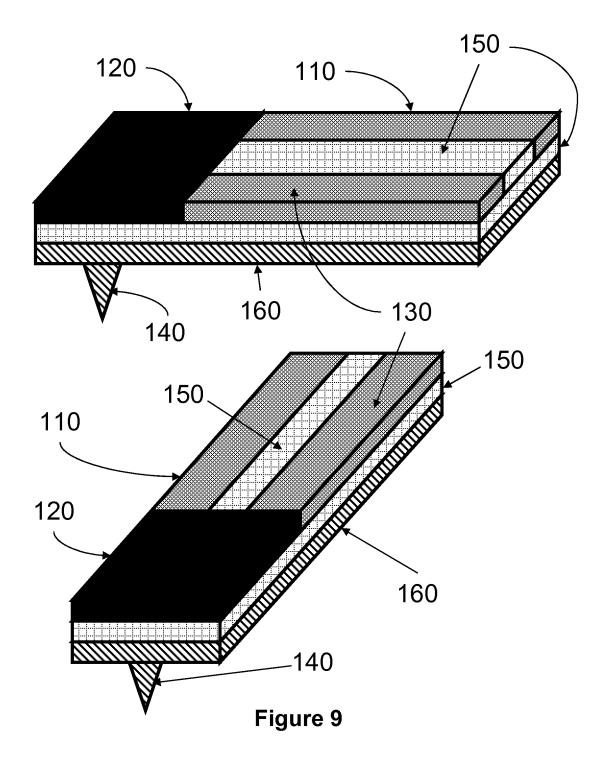


Figure 8



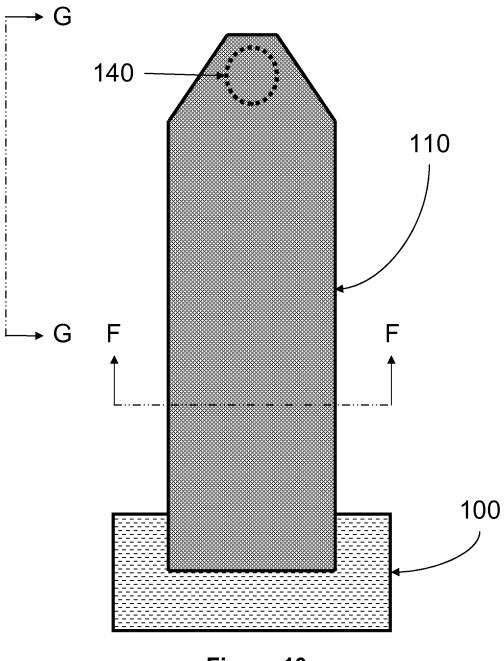


Figure 10

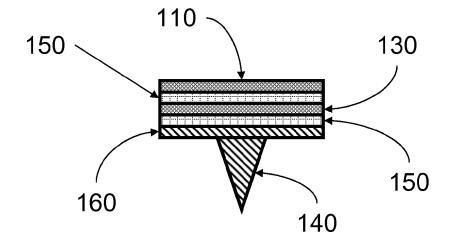


Figure 11

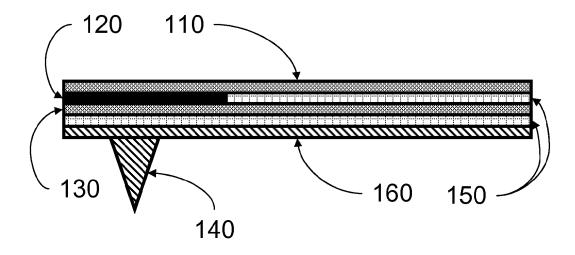
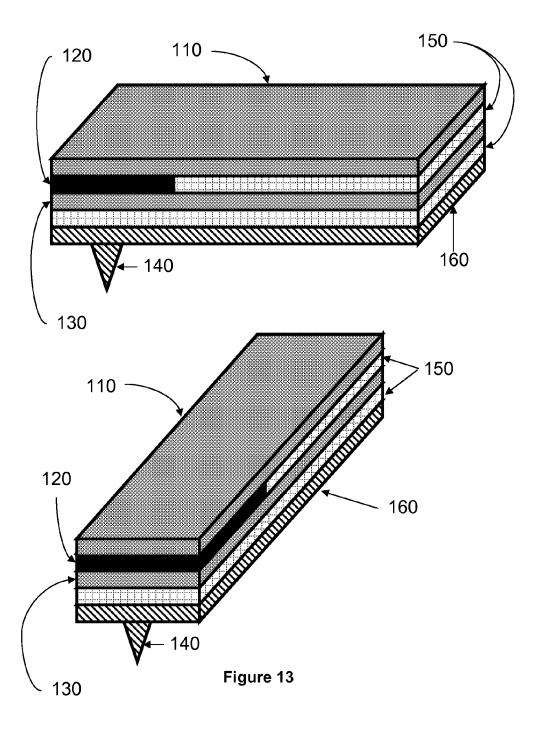


Figure 12



TEMPERATURE-DEPENDENT NANOSCALE CONTACT POTENTIAL MEASUREMENT TECHNIQUE AND DEVICE

CROSS REFERENCE TO RELATED APPLICATION

This application is a National Stage Application under 35 U.S.C. §371 of International Application No. PCT/US 09/32545, filed Jan. 30, 2009, which claims the benefit of and 10 priority to U.S. Provisional Application 61/024,962, filed on Jan. 31, 2008, which is hereby incorporated by reference in its entirety.

BACKGROUND OF THE INVENTION

This invention is in the field of atomic force microscope cantilevers. This invention relates generally to an atomic force microscope cantilever having an integrated heater-thermometer and conductive tip, useful for measuring or actuat- 20 ing temperature-dependant electrical potential with nanometer scale resolution.

Microcantilever devices having integrated resistive heaters have found use in the fields of microscopy and information describe microcantilevers having resistively heated tips used for patterning substrates for storage of binary information. Resistive heaters also find use in inducing mechanical oscillation for tapping mode atomic force microscopy measurements, as disclosed in U.S. Patent Application Publication 30 No. US2006/0238206.

In contrast, the microcantilever devices described herein, however, comprise heater-thermometers for controlling or measuring the temperature of a surface. The microcantilever devices described herein also comprise a conductive tip elec- 35 trically isolated from the resistive heater which allows for temperature dependent Kelvin probe microscopy measurements as well as other uses.

SUMMARY OF THE INVENTION

Provided herein are microcantilevers capable of and/or useful for independently measuring and/or controlling the electrical potential and/or temperature of a surface with nanometer scale position resolution. Also provided herein are 45 methods and devices for manipulating, imaging, and/or mapping a surface or the properties of a surface with a microcantilever. The microcantilevers described herein are also capable of and/or useful for independently measuring and/or controlling the electrical potential and/or temperature of a gas 50 or liquid. The devices and methods of the present invention are useful for applications including gas, liquid, and surface sensing, micro- and nano-fabrication, imaging and mapping of surface contours or surface properties.

In an embodiment, a microcantilever device of the present 55 invention comprises: a cantilever having a fixed end and a free end, a heater-thermometer positioned near the free end of the cantilever, and a conductive tip positioned near the free end of the cantilever, wherein the conductive tip is electrically isolated from the heater-thermometer. As used herein, the 60 expressions "heater-thermometer positioned near the free end of the cantilever" or "conductive tip positioned near the free end of the cantilever" refer to a relative position of the heaterthermometer or conductive tip between 0 and 200 µm of the cantilever free end, preferably for some applications between 65 0 and 50 μm or between 0 and 25 μm of the free end of the cantilever. This expression also includes embodiments where

2

at least a portion of the heater-thermometer or conductive tip is spatially coincident with the free end of the cantilever. The microcantilever devices of the present invention may further comprise one or more electrodes electrically connected to the heater-thermometer and/or the conductive tip.

In an embodiment, a microcantilever of the present invention is capable of producing and/or produces a temperature change in a surface. According to this aspect, a temperature change can be effected by bringing the tip of the microcantilever close to (e.g., within 500 nm or 1 µm), in thermal contact, in physical contact, or in electrical contact with the surface and providing a current to the heater-thermometer to heat a portion of the microcantilever adjacent to the tip. In this embodiment, the heater-thermometer and tip will reach a specified temperature which can be controlled and monitored; after this, the surface close to, in thermal contact, in physical contact, or in electrical contact with the tip will have thermal interaction with the tip, thereby producing a change in the temperature of the surface. In an exemplary embodiment, the heater-thermometer comprises a thermistor, such that the temperature can be monitored by measuring the resistance of the thermistor.

In another embodiment, a microcantilever of the present storage. For example, U.S. Pat. Nos. 6,762,402 and 7,038,996 25 invention is capable of measuring and/or measures the temperature of a surface. In embodiments where the microcantilever measures a surface temperature, the microcantilever tip is brought close to, in thermal contact, in physical contact, or in electrical contact with a surface and allowed to have thermal interaction with the surface. Subsequently, a signal from a temperature sensor near the tip of the microcantilever can be measured and the temperature determined. In an exemplary embodiment, the temperature sensor comprises a heater-thermometer. A heater-thermometer can be useful for simultaneously or independently controlling and/or sensing the temperature.

> In another aspect, a microcantilever is capable of producing and/or produces a change in the electrical potential of a 40 surface. In an embodiment of this aspect, the tip of the microcantilever is coated with and/or comprised of an electrically conductive material, and a voltage is provided to the tip, and thereby affects a change in the electrical potential of a surface when the tip is brought close to or in physical or electrical contact with the surface. In another embodiment, a microcantilever of the present invention is capable of sensing or measuring and/or senses or measures the electrical potential of a surface by bringing a conductive microcantilever tip close to or in physical or electrical contact with the surface and measuring the electrical potential of a the tip.

In an exemplary embodiment, a microcantilever of the present invention is independently capable of simultaneously measuring, sensing, and/or controlling the temperature and/ or electrical potential of a surface. For example, a microcantilever of a specific embodiment simultaneously controls the temperature of a surface while measuring the electrical potential of the surface, or simultaneously measures the temperature of a surface while controlling the electrical potential of the surface. In an exemplary embodiment, a microcantilever is capable of providing nanometer resolution mapping of the contours, height or profile of a surface, the temperature of a surface, the electrical potential of a surface, or any combination of these. In these embodiments, the microcantilever is fabricated with both a heater-thermometer and a conductive tip. In some embodiments, it is preferred that the conductive tip of the cantilever is electrically isolated from the heaterthermometer. In some embodiments, it is also preferred that

any leads electrically connected to the heater-thermometer are electrically isolated from any leads electrically connected to the conductive tip.

In another exemplary embodiment, a microcantilever is capable of providing and/or provides nanometer resolution 5 mapping of the contours, height or profile of a surface as a function of temperature while simultaneously mapping the electrical potential of the surface as a function of temperature. In embodiments where the contours, height or profile of the surface are mapped, methods well known in the art of atomic 10 force microscopy and/or scanning tunneling microscopy can be used. Methods for mapping the contour profile of a surface include, but are not limited to: using a laser spot reflected from the top of the cantilever into an array of photodiodes, optical interferometry, capacitive sensing, piezoresistive or 15 piezoelectric sensors within the cantilever, measurement of tunneling current, or any combination of these or other methods useful for sensing the surface profile.

In another aspect, provided is a method of sensing an attribute of a surface, the method comprising the steps of: 20 providing a surface; providing a microcantilever device of the present invention having a conductive tip close to, in thermal contact, or in physical contact with the surface; allowing the conductive tip to have thermal interaction with the surface; and measuring an electrical property of the conductive tip or 25 a heater-thermometer of the microcantilever, thereby sensing an attribute of the surface. Useful electrical properties to sense an attribute of a surface comprise the resistance across the heater thermometer, and the electrical potential of the conductive tip. In an exemplary embodiment, attributes 30 capable of being sensed comprise the temperature of the surface, the electrical potential of the surface, and both the temperature and electrical potential of the surface. In an embodiment, the temperature of the surface can be sensed by measuring a resistance of the heater-thermometer. In an 35 embodiment, the electrical potential of the surface is sensed by measuring a voltage or electric potential of a conductive tip of the microcantilever device.

In another aspect, the present invention provides a method of controlling an attribute of a surface, the method comprising 40 the steps of: providing a surface; providing a microcantilever device of the present invention having a conductive tip close to, in thermal contact, in physical contact, or in electrical contact with the surface; providing a voltage and/or current to the conductive tip or a heater-thermometer of the microcantilever device, and allowing the surface to have thermal interaction with one or more portions of the microcantilever device.

Surface attributes useful for controlling with the methods described herein comprise the temperature of the surface, the selectric potential of the surface, and both the temperature and electric potential of the surface. In an embodiment, control of the temperature is controlled by providing a current to a heater-thermometer portion of the microcantilever device. In an embodiment, control of the electrical potential of the surface is achieved by providing a voltage to a conductive tip of the microcantilever device.

In another aspect, the present invention also provides a method of manipulating a surface, the method comprising: providing a surface; providing a microcantilever device of the 60 present invention close to, in thermal contact, in physical contact, or in electrical contact with the surface; and providing a voltage and/or current to a conductive tip and/or heater-thermometer of the microcantilever device. This method may optionally further comprise: allowing the surface to have 65 thermal interaction with the heater-thermometer portion of the microcantilever device and providing a second current

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and/or voltage to the conductive tip and/or heater-thermometer microcantilever device. In an exemplary embodiment, the first voltage and/or current results in a temperature change of the heater-thermometer portion of the microcantilever device and/or the surface and the second voltage and/or current provided results in a manipulation of the surface, effectively manipulating the surface at a fixed and/or controlled temperature.

Surface manipulations useful with the methods described herein comprise: changing the physical state of the surface, for example from a solid to a liquid or a gas; heating the surface; cooling the surface; changing or inducing a magnetic orientation of the surface; changing the level of oxidation of the surface; creating a glass transition in the surface; injecting electrons into the surface; driving a current into or through the surface; depositing material onto the surface; and any combination of these or other useful surface manipulations.

The microcantilever devices of some embodiments can also be used to sense or control the temperature and/or electrical potential of a liquid or a gas surrounding the microcantilever device. In an embodiment, a microcantilever of the present invention is capable of producing and/or produces a temperature change in a liquid or a gas or is capable of determining and/or determines the temperature of a liquid or a gas. According to this aspect, a temperature change can be affected by bringing the tip of the microcantilever, in thermal contact, in physical contact, or in electrical contact with the liquid or gas and providing a current or voltage to the heaterthermometer to heat a portion of the microcantilever adjacent to the tip. In this embodiment, the heater-thermometer and tip will reach a specified temperature which can be controlled and monitored; after this, the liquid or gas in thermal contact, in physical contact, or in electrical contact with the tip will have thermal interaction with the tip, thereby producing a change in the temperature of the liquid or gas. In embodiments where the temperature of the liquid or the gas is determined, the tip of the microcantilever is brought into thermal, physical, or electrical contact with the liquid or the gas and allowed to have thermal communication with the liquid or the gas. Subsequently the temperature of the liquid or gas is determined by measuring an electrical property of the heaterthermometer.

In another embodiment, a microcantilever of the present invention is capable of producing and/or produces a change in or is capable of determining and/or determines the electrical potential of a liquid or a gas. According to this aspect, a change in the electrical potential can be effected by bringing the tip of the microcantilever into thermal contact, physical contact, or electrical contact with the liquid or gas and providing a current or voltage to the conductive tip. In embodiments where the electrical potential of the liquid or the gas is determined, the tip of the microcantilever is brought into thermal contact, physical contact, or electrical contact with the liquid or gas and the voltage of the conductive tip subsequently determined.

In an exemplary embodiment, the present invention provides a method of manipulating a surface. A method of this aspect comprises providing a surface, providing a microcantilever device of the present invention having a conductive tip and a heater-thermometer in thermal, physical, or electrical contact with a gas or liquid between or near the surface and the microcantilever, and providing a voltage or current to the heater-thermometer, the conductive tip, or both. In some embodiments, the temperature or electrical potential of the liquid or gas is changed by the voltage or current provided to the heater-thermometer, the conductive tip, or both, and the gas or liquid having undergone a temperature or electrical

potential change subsequently reacts with the surface, thereby manipulating or modifying the surface. In another embodiment, the current or voltage provided to the heater-thermometer, the conductive tip, or both cause a discharge from the conductive tip to the liquid or gas. Subsequently, the gas or liquid present in the discharged region undergoes a chemical or physical reaction with the surface, thereby modifying the surface.

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

FIG. 1 provides example data showing that the tip temperature can be calibrated as a function of the electrical resistance. $_{\rm 20}$

FIG. 2 provides a schematic showing an overhead view of a first embodiment of a microcantilever device.

FIG. 3 provides a schematic showing a front view of a first embodiment of a microcantilever device.

FIG. **4** provides a schematic showing a side view of a first 25 embodiment of a microcantilever device.

FIG. 5 provides a schematic showing a side view of a first embodiment of a microcantilever device.

FIG. 6 provides a schematic showing an overhead view of a second embodiment of a microcantilever device.

FIG. 7 provides a schematic showing a rear view of a second embodiment of a microcantilever device.

FIG. 8 provides a schematic showing a side view of a second embodiment of a microcantilever device.

FIG. **9** provides perspectives views of a second embodi- ³⁵ ment of a microcantilever device.

FIG. 10 provides a schematic showing an overhead view of a third embodiment of a microcantilever device.

FIG. 11 provides a schematic showing a rear view of a third embodiment of a microcantilever device.

FIG. 12 provides a schematic showing a side view of a third embodiment of a microcantilever device.

FIG. 13 provides perspective views of a third embodiment of a microcantilever device.

DETAILED DESCRIPTION OF THE INVENTION

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 50 skilled in the art. The following definitions are provided to clarify their specific use in the context of the invention.

"Heater-thermometer" refers to a combination of a device for determining temperature and a device for actuating the temperature. In an embodiment, a thermistor is useful as a 55 heater-thermometer. A thermistor refers to a resistive material which has a resistance which is temperature dependant. Providing a current or voltage to a thermistor can result in an increase in the temperature of the thermistor through resistive heating. Since the resistance of a thermistor is temperature dependent, it can be used as means for measuring the temperature; i.e., by measuring the resistance of the thermistor, the temperature of the thermistor can be determined. A thermistor useful with some embodiments of the present invention comprises doped silicon, for example silicon doped with 65 a phosphorus concentration of about 1×10^{17} cm⁻³. A heater-thermometer can refer to a single or separate distinct elements

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for measuring and actuating the temperature, for example a thermistor or a thermocouple and a resistive heater.

"Thermal steady state" refers to a condition of a material or element at which the temperature of the material or element is substantially constant, for example a condition where the temperature changes at a rate of less than 5 K/minute. Thermal steady state can also refer to a condition of thermal equilibrium or a condition where the heat input is substantially equal to the heat losses and/or heat output.

"Thermal communication" and "thermal contact" refers to an orientation or position of elements or materials, such as a heater-thermometer and a conductive tip, such that there is more efficient transfer of heat between the two elements than if isolated or thermally insulated. Elements or materials may
 be considered in thermal communication or contact if heat is transported between them more quickly than if they were isolated or thermally insulated. Two elements in thermal communication or contact may reach thermal equilibrium or thermal steady state and in some embodiments may be considered to be constantly at thermal equilibrium or thermal steady state with one another. In some embodiments, elements in thermal communication with one another may be separated by a distance of 1 μm or less.

"Thermal insulation" refers to a material that is used to reduce the rate of heat transfer. In one aspect, thermal insulation reduces the rate of heat transfer between elements or materials to a rate less than the rate when the elements or materials are in physical contact.

"Electrical isolation" refers to elements or materials which are not electrically connected, in electrical contact, or in electrical communication. In one aspect, electrical isolation can be provided by physical separation of elements or materials, i.e. the elements or materials are not in physical contact. In this aspect, the elements or materials may be spatially separated or separated by an electrically insulating material. In another aspect, electrical isolation can be provided by a difference in the electrical properties of a material or element. For example, a metal or conductive material can be considered electrically isolated from a non-conductive or electri-40 cally insulating material even though the materials are in physical contact. Due to a difference in electrical properties, conducting materials may be considered to be in electrical isolation from one another even if they are in physical contact; for example, since current is permitted to flow in only one direction across a diode junction, the two sides of a diode may be considered to be electrically isolated from one another when considered in one direction and may be considered to be electrically connected to one another when considered in the opposite direction.

"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 $1000\,\mu m$. The cantilevers useful in the present invention include, but are not limited to, cantilevers having any useful shape, including platform or rectangular shaped cantilevers, circular shaped cantilevers, ladder shaped cantilevers, U-shaped cantilevers, serpentine shaped cantilevers, and cantilevers having cutout portions.

In one aspect, the present invention provides a microcantilever device useful for investigating properties of a surface, as well as for making modifications to a surface. In another aspect, the present invention provides methods of probing, sensing, or controlling the properties of a surface, such as the temperature or electrical potential of a surface. In yet another aspect, the present invention provides methods of modifying

surfaces, such as selectively depositing material onto a surface, selectively heating regions of a surface, selectively changing the physical state of regions of a surface, selectively cooling regions of a surface, selectively changing or inducing a magnetic orientation of a region of a surface, selectively changing the level of oxidation of a region of a surface, selectively creating a glass transition in a region of a surface, or selectively injecting electrons into a region of surface. In the context of this description, the term "selectively" refers to processes wherein a portion or region of a surface having a selected position is manipulated. The microcantilever devices of some embodiments are useful for probing and modifying surfaces with nanometer scale resolution.

In an embodiment, a device of the present invention comprises a cantilever having a fixed end and a free end. Useful 15 cantilevers include those having any shape. In general, cantilevers are capable of being manufactured in a variety of ways, including methods known in the art of silicon-on-insulator (SOI) fabrication. Exemplary cantilevers useful in the present invention comprise crystalline or polycrystalline sili- 20 con. Cantilevers useful in some embodiments of the present invention may also comprise one or more regions of doped semiconductor, such as phosphorus or boron doped silicon, or n-type or p-type silicon, or doped diamond, or one or more regions of an insulating material, such as silicon oxide or 25 silicon nitride. Cantilevers of the present invention are capable of being used in an atomic force or other type of surface probe microscope. Cantilevers of embodiments of the present invention are capable of being constructed in a variety of forms, including cantilevers having one or more supporting 30 legs.

In an embodiment, a device of the present invention comprises one or more electrodes positioned along a cantilever. In some embodiments, the electrodes comprise one or more legs of a cantilever. In an exemplary embodiment, electrodes use- 35 ful in the present invention comprise doped semiconductor, for example doped silicon or doped diamond. In other exemplary embodiments, electrodes useful in the present invention comprise a metal, for example tungsten, gold, aluminum, platinum, nickel, or any other metal. In some embodiments, 40 an electrode may comprise a metal coating. In some applications, doped semiconductor is preferred over metallic electrodes since doped semiconductor may be capable of withstanding higher temperatures where some metals will melt, for example temperatures up to 1250° C. Doped semiconduc- 45 tor may also be preferred for some applications since it may be capable of supporting a higher current density than a metal electrode of similar dimensions. In other applications, metallic electrodes may be preferred over doped semiconductor, due to the relatively lower electrical resistance of many metals. The level of doping in doped semiconductor, however, can be selectively adjusted to create regions of doped semiconductor having higher or lower resistances. Regions of doped semiconductor having low resistances can be useful as electrodes or electrical interconnections. Regions of doped 55 semiconductor having higher resistances can be useful as resistive heaters or thermistors or heater-thermometers.

In an embodiment, a device of the present invention also comprises a heater-thermometer, positioned near the free end of a cantilever. In an exemplary embodiment, a heater-thermometer is capable of heating and/or heats the end of a cantilever on which it is integrated as well as the surface. Heat may be produced in a heater-thermometer by providing a current and/or voltage to the heater-thermometer or to electrodes electrically connected to the heater-thermometer; in 65 this way, the heater-thermometer can be resistively heated. Useful heater-thermometers are also capable of determining

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and/or determine the temperature of a cantilever. In an embodiment, by determining the resistance of the heater-thermometer, the temperature of the heater-thermometer and/or cantilever can be determined to high precision, for example to a precision of 5° C. or, more preferably, 1° C. FIG. 1 provides data showing results of Raman microscopy calibration of the cantilever temperature and indicate that the temperature can be calibrated as a function of the electrical resistance of the heater-thermometer, in this case with a precision of 5° C.

In an embodiment, a device of the present invention is also comprised of a conductive tip positioned near the free end of a cantilever. Useful tips include tips which are capable of use in an atomic force microscope, such as a tip with a very small radius of curvature. In an embodiment preferred for some applications, the conductive tip is comprised of a metal or a metal coating. In another embodiment, the conductive tip is electrically connected to an electrode portion of the cantilever. In an embodiment, the conductive tip is comprised of a hard, patternable metal, for example aluminum, gold, tungsten, platinum, or nickel. In another embodiment, a conductive tip is comprised of a doped semiconductor, for example doped diamond or doped silicon or other doped semiconductor. In a preferred embodiment, the conductive tip and any electrode to which it is electrically connected are comprised of the same material. In addition to other uses, a conductive tip can be useful for microscopy, nanolithography, and dippen nanolithography. A conductive tip is also useful for probing, sensing, or controlling the potential of a surface, for example by providing or measuring a voltage the conductive tip or of an electrode electrically connected to the conductive

In a preferred embodiment, a conductive tip is electrically isolated from other components of the cantilever, for example a heater-thermometer and any electrodes electrically connected to the heater-thermometer. Electrical isolation may be provided in a variety of methods, including physical separation or use of a layer of an electrically insulating material between the conductive tip and heater-thermometer. Useful electrically insulating materials include, but are not limited to, undoped silicon, silicon oxide, silicon nitride, diamond, and polymers. In some embodiments the electrically insulating material is thermally conductive and allows for thermal communication between the conductive tip and the heaterthermometer; in other embodiments, the electrically insulating material also provides thermal insulation between the conductive tip and the heater-thermometer. In an embodiment, a conductive tip is in thermal communication with a heater-thermometer on the same cantilever, whereby the conductive tip has a temperature within 10 K of that of the heater-thermometer. In some embodiments, the conductive tip and heater-thermometer may be spatially offset from one another; in other embodiments, the conductive tip and heaterthermometer may be adjacent to or on top of one another.

Referring now to the drawings, FIG. 2 shows a first preferred embodiment of a microcantilever device of the present invention. In this embodiment, the microcantilever is supported by a holder chip 100 which may be patterned to provide electrical connections to various electrical leads on the microcantilever. In this embodiment, a first electrode 110 is comprised of highly doped silicon such that it has a relatively low resistance, for example a resistance less than 10% of the resistance of the heater-thermometer 120. In this embodiment, first electrode 110 comprises one leg of the cantilever. Electrically connected to first electrode 110 is heater-thermometer 120 near the free end of the cantilever, which, in some embodiments, is comprised of doped silicon such that it

has a higher resistance than the first electrode 110 or a second electrode 130. In this embodiment, second electrode 130 is also comprised of highly doped silicon such that it has a relatively low resistance. On the bottom of the free end of the cantilever there is a conductive tip 140. In this embodiment, conductive tip 140 resides partially on insulating material 150 which provides electrical isolation between conductive tip 140 and heater-thermometer 120. Insulating material 150 also provides electrical isolation between heater-thermometer 120 and third electrode 160. Comprising part of the free end of the cantilever, as well as a leg of the cantilever, is third electrode 160, which is comprised of a metal coating on the silicon substrate. Third electrode 160 is electrically connected to conductive tip 140.

FIG. 3 shows a view of the first preferred embodiment in the A-A direction. Heater-thermometer 120 is partially shown and attached to insulating material 150 which provides electrical isolation between conductive tip 140 and third electrode 160. FIG. 4 shows a view of the first preferred embodiment in the B-B direction, showing conductive tip 140 and third electrode 160. FIG. 5 shows a view of the first preferred embodiment in the C-C direction, showing first electrode 110, heater-thermometer 120, insulating material 150 and conductive tip 140. The first embodiment shows the heater-thermometer and conductive tip spatially offset from one another, and also shows the cantilever as a single layer; however, cantilevers of the present invention can comprise multiple layers.

FIG. **6** shows a second preferred embodiment of a microcantilever device of the present invention comprising multiple layers. In this embodiment, the topmost layer is comprised of first electrode **110**, heater-thermometer **120**, second electrode **130**, and insulating material **150**. FIG. **7** shows a cross sectional view of the second preferred embodiment in the D-D direction, and shows that the topmost layer is separated from the bottommost layer, which comprises third electrode **140**, by a middle layer comprising insulating material **150**. FIG. **8** shows a view of the second preferred embodiment in the E-E direction, showing that heater-thermometer **120** is located on top of conductive tip **140**. FIG. **9** shows perspective views of an additional interpretation of the second preferred embodiment.

FIG. 10 shows a third preferred embodiment of a microcantilever device of the present invention comprising multiple layers. In this embodiment, the topmost layer is comprised substantially of first electrode 110. FIG. 11 shows a cross sectional view of the third preferred embodiment in the F-F direction and FIG. 12 shows a view in the G-G direction. Here, the topmost layer comprises first electrode 110. The next layer is partially comprised of insulating material 150 and partially comprised of heater-thermometer 120. In this embodiment, heater-thermometer 120 is located above conductive tip 140. The next layer is comprised of second electrode 130 which is located above a second insulating material 150. The bottommost layer in this embodiment comprises third electrode 160. FIG. 13 shows perspective views of an additional interpretation of the third preferred embodiment.

It will be appreciated from the foregoing that microcantilever devices of the present invention can be constructed in many different embodiments. The preferred embodiments described above are not intended to limit the invention which is defined by the following claims.

The invention may be further understood by the following non-limiting examples.

EXAMPLE 1

Design and Fabrication of a Microcantilever Having an Integrated Electrode and Heater Element

The AFM cantilevers of the present invention comprise an integrated resistive heating element and an electrically-ad-

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dressable metal-coated tip. The resistive heater is capable of reaching temperatures exceeding 1000° C. The microcantilevers of the present invention are calibrated such that the tip temperature can be controlled to within 1° C. Ideal cantilevers have a spring constant in the range 0.1-1 N/m and have a resonant frequency in the range of 30 kHz. These cantilevers can be used in either tapping mode or contact mode operation.

FIG. 2 shows a schematic of an exemplary cantilever of the present invention. The cantilever is made of single-crystal silicon. Some of the silicon is doped, in a process described below, in order to achieve cantilever heating. The cantilever has three legs—two of the legs are made of heavily doped silicon to carry electrical current, and the third leg is made of metal-coated silicon. The metal electrode of the third leg extends to the tip and coats the tip at the end of the cantilever. The electrical potential at the end of the sharp tip can be read from the electrode leg. The doped silicon legs carry current to the heater region near the free end of the cantilever. The heater region of the cantilever is made of doped silicon. The legs have a higher doping concentration than the heater region. such that the legs are highly conducting and the heater region is somewhat more resistive. Doped silicon legs are preferred for delivery of current to the heater region rather than metal, because a metal leg would not be able to carry the current required for heating without exceeding its current density limit. The metal electrode is selected for the electrical potential measurement, and is preferred over doped silicon because it has very low electrical resistivity.

The heated AFM cantilevers are fabricated using a standard silicon-on-insulator (SOI) process known in the art, but modified to accommodate the electrode required for the Kelvin Probe measurements. The fabrication process starts with a SOI wafer of orientation <100>, n-type doping at 2×10^{14} cm⁻³ having a resistivity of approximately 4Ω -cm. The cantilever tips are formed using an oxidation sharpening process, which can achieve a tip radius of curvature of 20 nm or smaller. The silicon of the cantilever is made electrically active through two phosphorous doping steps: first, two parallel cantilever legs are doped to 1×10^{20} cm⁻³ and the heater region near the free end of the cantilever is doped to 1×10^{17} cm⁻³. The heater region is more resistive than the rest of the cantilever, such that when electrical current flows through the legs of the cantilever, heating occurs primarily in the highly resistive region near the free end of the cantilever. With the cantilever dimensions and temperature-dependent resistivities well defined, the cantilever electrical resistance depends on the cantilever temperature solely in the heater region to within 10%. Finally, the cantilever metal is patterned to form the tip electrode and the electrical connections to the doped silicon.

Metals for the tip electrode are selected from the group comprising tungsten, gold, and aluminum, platinum, nickel, and any other hard, patternable metal useful for standard deposition and microfabrication processes. These metals are useful because the metal is deposited and patterned using standard microfabrication processes, and must be sufficiently hard such that it does not deform considerably during scanning contact with a hard surface. The work function of the metal coating is also an important consideration for potential measurements.

The cantilever temperature can be calibrated using infrared (IR) and Raman microscopy. FIG. 1 shows results of Raman microscopy calibration of the cantilever temperature, in this case with a precision of 5° C. The cantilever electrical resistance depends very strongly upon temperature and thus it is possible to control the cantilever tip temperature by monitoring the cantilever electrical resistance.

In the cantilever schematic of FIG. 2, the cantilever heater is spatially offset from the metal electrode. As the tip and heater are in thermal contact, the tip temperature can be calibrated even though it is offset from the heater. Alternatively, the heater can be located directly on top of the tip, as shown in FIGS. 6-9, in which case the electrode is electrically isolated from the heater, for example by a thin film passivation layer such as silicon dioxide.

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STATEMENTS REGARDING INCORPORATION BY REFERENCE AND VARIATIONS

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).

All patents and publications mentioned in the specification 50 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 55 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 60 referenced patent documents), are not intended to be included in the claim.

When a group of substituents is disclosed herein, it is understood that all individual members of those groups and all subgroups, and classes that can be formed using the substituents are disclosed separately. When a Markush group or other grouping is used herein, all individual members of the 12

group and all combinations and subcombinations possible of the group are intended to be individually included in the disclosure.

Every formulation or combination of components described or exemplified can be used to practice the invention, unless otherwise stated. Specific names of compounds or materials are intended to be exemplary, as it is known that one of ordinary skill in the art can name the same compounds or materials differently. One of ordinary skill in the art will 10 appreciate that methods, device elements, starting materials, and synthetic and fabrication methods other than those specifically exemplified can be employed in the practice of the invention without resort to undue experimentation. All artknown functional equivalents, of any such methods, device elements, starting materials, and synthetic and fabrication 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.

As used herein, "comprising" is synonymous with "including," "containing," 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.

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.

I claim:

- 1. A device for measuring or controlling temperature-dependent electrical properties of a surface comprising:
 - a. a cantilever having a fixed end and a free end, said cantilever comprising single crystal silicon;
 - b. a heater-thermometer positioned near said free end of said cantilever, wherein said heater-thermometer comprises a thermistor comprising doped single crystal silicon;
- c. a conductive tip electrode for measuring or controlling a voltage between said conductive tip electrode and said surface or for flowing a current between said conductive tip electrode and said surface, said conductive tip electrode positioned near said free end of said cantilever, wherein said conductive tip electrode is electrically isolated from said heater-thermometer;

- d. a layer of insulating material positioned to provide electrical isolation between said conductive tip electrode and said heater-thermometer; and
- e. one or more single crystal silicon electrodes electrically connected to said heater-thermometer or said conductive tip electrode, wherein said one or more single crystal silicon electrodes comprise one or more legs of said cantilever.
- 2. The device of claim 1 wherein said one or more single crystal silicon electrodes comprise a first doped single crystal silicon electrode electrically connected to said heater-thermometer, a second doped single crystal silicon electrode electrically connected to said heater-thermometer, and a third electrode electrically connected to said conductive tip electrode
- 3. The device of claim 2 wherein said third electrode comprises doped single crystal silicon or metal coated single crystal silicon.
- 4. The device of claim 1 wherein said one or more single 20 crystal silicon electrodes comprise a first doped single crystal silicon electrode electrically connected to said heater-thermometer, and a second electrode electrically connected to both said heater-thermometer and said conductive tip electrode.
- 5. The device of claim 4 wherein said second electrode comprises doped single crystal silicon or metal coated single crystal silicon.
- 6. The device of claim 1 wherein said one or more electrodes deliver electrical current to said heater-thermometer 30 for effecting a temperature change in said heater-thermometer
- 7. The device of claim 1 wherein said one or more electrodes provide a voltage or current to or from said conductive tip electrode.
- **8**. The device of claim **1** wherein said one or more electrodes comprise a material that can withstand a temperature up to 1250° C.
- 9. The device of claim 1 wherein said one or more electrodes comprise a metal.
- 10. The device of claim 9 wherein said metal is selected from the group consisting of tungsten, gold, aluminum, platinum, and nickel.
- 11. The device of claim 1 wherein said heater-thermometer comprises a material that can withstand a temperature up to 451250° C.
- 12. The device of claim 1 wherein said conductive tip electrode comprises a metal or a doped semiconductor.
- 13. The device of claim 12 wherein said metal is selected from the group consisting of tungsten, gold, aluminum, platinum, and nickel.
- **14**. The device of claim **12** wherein said doped semiconductor is selected from the group consisting of doped diamond and doped silicon.
- **15**. The device of claim **1** wherein the layer of insulating 55 material is selected from the group consisting of undoped silicon, silicon oxide, silicon nitride, diamond, and polymer.
- 16. The device of claim 1 wherein said electrical isolation also provides thermal insulation between the conductive tip electrode and said heater-thermometer.
- 17. The device of claim 1 wherein said electrical isolation allows for thermal communication between the conductive tip electrode and said heater-thermometer.
- **18**. The device of claim **1** wherein said heater-thermometer is spatially offset from said conductive tip electrode.
- 19. The device of claim 1 wherein said heater-thermometer is located on top of said conductive tip electrode.

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- 20. The device of claim 1, wherein said conductive tip electrode comprises a conductive coating over a cantilever tip.
- 21. A method of sensing an attribute of a surface, a liquid, or a gas, the method comprising:
 - a. providing said surface, said liquid, or said gas;
 - b. providing a device having a conductive tip electrode in thermal or electrical communication with said surface, said liquid, or said gas, said device comprising:
 - i. a cantilever having a fixed end and a free end, said cantilever comprising single crystal silicon;
 - ii. a heater-thermometer positioned near said free end of said cantilever, wherein said heater-thermometer comprises a thermistor comprising doped single crystal silicon;
 - iii. said conductive tip electrode for measuring or controlling a voltage between said conductive tip electrode and said surface, said liquid, or said gas or for flowing a current between said conductive tip electrode and said surface, said liquid, or said gas, said conductive tip electrode positioned near said free end of said cantilever, wherein said conductive tip electrode is electrically isolated from said heater-thermometer;
 - iv. a layer of insulating material positioned to provide electrical isolation between said conductive tip electrode and said heater-thermometer; and
 - v. one or more single crystal silicon electrodes electrically connected to said heater-thermometer or said conductive tip electrode, wherein said one or more single crystal silicon electrodes comprise one or more legs of said cantilever; and
 - measuring an electrical property of said heater-thermometer, said conductive tip electrode, or both, thereby sensing said attribute of said surface, said liquid, or said gas
- 22. The method of claim 21 wherein said attribute of said surface, said liquid, or said gas is selected from the group consisting of the temperature of said surface, said liquid, or said gas; the electrical potential of said surface, said liquid, or said gas; and both the temperature and electrical potential of said surface, said liquid or said gas.
- 23. The method of claim 21 wherein said temperature of said surface, said liquid, or said gas is sensed by measuring a resistance across said heater-thermometer.
- 24. The method of claim 21 wherein said electrical potential of said surface, said liquid, or said gas is sensed by measuring a voltage of said conductive tip electrode.
- 25. The method of claim 21 wherein in step c said electrical property is selected from the group consisting of the resistance across said heater-thermometer; and the electrical potential of said conductive tip electrode.
- **26**. A method of controlling an attribute of a surface, a liquid, or a gas, the method comprising:
 - a. providing said surface, said liquid, or said gas;
 - b. providing a device having a conductive tip electrode in thermal or electrical communication with said surface, said liquid, or said gas, said device comprising:
 - i. a cantilever having a fixed end and a free end, said cantilever comprising single crystal silicon;
 - ii. a heater-thermometer positioned near said free end of said cantilever, wherein said heater-thermometer comprises a thermistor comprising doped single crystal silicon;
 - iii. said conductive tip electrode for controlling or measuring a voltage between said conductive tip electrode and said surface, said liquid, or said gas or for flowing

- a current between said conductive tip electrode and said surface, said liquid, or said gas, said conductive tip electrode positioned near said free end of said cantilever, wherein said conductive tip electrode is electrically isolated from said heater-thermometer; 5
- iv. a layer of insulating material positioned to provide electrical isolation between said conductive tip electrode and said heater-thermometer; and
- v. one or more single crystal silicon electrodes electrically connected to said heater-thermometer or said conductive tip electrode, wherein said one or more single crystal silicon electrodes comprise one or more legs of said cantilever; and
- c. providing a voltage or current to said heater-thermometer, said conductive tip electrode, or both.
- 27. The method of claim 26 wherein said attribute of said surface, said liquid, or said gas is selected from the group consisting of the temperature of said surface, said liquid, or 20 said gas; the electrical potential of said surface, said liquid, or said gas; and both the temperature and electrical potential of said surface, said liquid, or said gas.
- 28. The method of claim 26 wherein said temperature of said surface, said liquid, or said gas is controlled by providing 25 a current to said heater-thermometer.
- 29. The method of claim 26 wherein said electrical potential of said surface, said liquid, or said gas is controlled by providing a voltage to said conductive tip electrode.
- 30. A method of manipulating a surface, said method comprising:
 - a. providing a surface;
 - b. providing a device having a conductive tip electrode in thermal or electrical communication with said surface, said device comprising:
 - i. a cantilever having a fixed end and a free end, said cantilever comprising single crystal silicon;
 - ii. a heater-thermometer positioned near said free end of said cantilever, wherein said heater-thermometer comprises a thermistor comprising doped single crys-40 tal silicon:
 - iii. said conductive tip electrode for measuring or controlling a voltage between said conductive tip electrode and said surface or for flowing a current between said conductive tip electrode and said surface, said 45 conductive tip electrode positioned near said free end of said cantilever, wherein said conductive tip electrode is electrically isolated from said heater-thermometer; and
 - iv. a layer of insulating material positioned to provide 50 electrical isolation between said conductive tip electrode and said heater-thermometer; and
 - v. one or more single crystal silicon electrodes electrically connected to said heater-thermometer or said single crystal silicon electrodes comprise one or more legs of said cantilever; and
 - c. providing a voltage or current to said heater-thermometer, said conductive tip electrode, or both.
- said surface is selected from the group consisting of changing the physical state of said surface; heating said surface; cooling said surface; changing or inducing a magnetic orientation of said surface; changing the level of oxidation of said surface; creating a glass transition in said surface; injecting 65 electrons into said surface; and depositing material onto said surface.

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- 32. The method of claim 30 further comprising
- d. allowing said surface to have thermal interaction with said heater-thermometer; and
- e. providing a second voltage or current to said heaterthermometer, said conductive tip electrode, or both.
- 33. The method of claim 32 wherein the current or voltage provided in step c results in a temperature change of said heater-thermometer and said surface and wherein said second voltage or current provided in step e effects a manipulation of said surface selected from the group consisting of changing the physical state of said surface; heating said surface; cooling said surface; changing or inducing a magnetic orientation of said surface; changing the level of oxidation of said surface; creating a glass transition in said surface; injecting 15 electrons into said surface; and depositing material onto said
 - 34. A method of manipulating a surface, said method comprising:
 - a. providing a surface;
 - b. providing a device having a conductive tip electrode in thermal or electrical communication with a gas or a liquid, said gas or said liquid positioned between said surface and said conductive tip electrode, said device comprising:
 - i. a cantilever having a fixed end and a free end, said cantilever comprising single crystal silicon;
 - ii. a heater-thermometer positioned near said free end of said cantilever, wherein said heater-thermometer comprises a thermistor comprising doped single crys-
 - iii. said conductive tip electrode for measuring or controlling a voltage between said conductive tip electrode and said surface, said liquid, or said gas or for flowing a current between said conductive tip electrode and said surface, said liquid, or said gas, said conductive tip electrode positioned near said free end of said cantilever, wherein said conductive tip electrode is electrically isolated from said heater-thermometer; and
 - iv. a layer of insulating material positioned to provide electrical isolation between said conductive tip electrode and said heater-thermometer; and
 - v. one or more single crystal silicon electrodes electrically connected to said heater-thermometer or said conductive tip electrode, wherein said one or more single crystal silicon electrodes comprise one or more legs of said cantilever; and
 - c. providing a voltage or current to said heater-thermometer, said conductive tip electrode, or both.
 - 35. The method of claim 34 wherein the current or voltage provided in step c results in a temperature change of said heater-thermometer and said gas or said liquid, or a discharge from said conductive tip electrode to said gas or said liquid.
- **36**. The method of claim **35** wherein said temperature conductive tip electrode, wherein said one or more 55 change of said liquid or said gas or said discharge from said conductive tip electrode to said liquid or said gas results in a chemical or physical reaction of said surface with said gas or said liquid.
- 37. A device for measuring or controlling temperature-31. The method of claim 30 wherein said manipulation of 60 dependent electrical properties of a surface, a liquid, or a gas comprising:
 - a. a cantilever having a fixed end and a free end, said cantilever comprising single crystal silicon;
 - b. a heater-thermometer positioned between 0 and 200 µm of said free end of said cantilever, wherein said heaterthermometer comprises a thermistor comprising doped single crystal silicon;

- c. a conductive tip electrode for measuring or controlling a voltage between said conductive tip electrode and said surface, said liquid, or said gas or for flowing a current between said conductive tip electrode and said surface, said liquid, or said gas, said conductive tip electrode positioned between 0 and 200 µm of said free end of said cantilever, wherein said conductive tip electrode is electrically isolated from said heater-thermometer;
- d. a first doped single crystal silicon electrode electrically connected to said heater-thermometer;
- e. a second doped single crystal silicon electrode electrically connected to said heater-thermometer;
- f. a third electrode electrically connected to said conductive tip electrode; and
- g. a layer of insulating material positioned to provide electrical isolation between said conductive tip electrode and said heater-thermometer;

wherein one or more of said first doped single crystal silicon electrode, said second doped single crystal silicon electrode 20 and said third electrode comprise one or more legs of said cantilever.

38. A device for measuring or controlling temperature-dependent electrical properties of a surface, a liquid, or a gas comprising:

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- a. a cantilever having a fixed end and a free end, said cantilever comprising single crystal silicon;
- b. a heater-thermometer positioned between 0 and 200 µm of said free end of said cantilever, wherein said heaterthermometer comprises a thermistor comprising doped single crystal silicon;
- c. a conductive tip electrode for measuring or controlling a voltage between said conductive tip electrode and said surface, said liquid, or said gas or for flowing a current between said conductive tip electrode and said surface, said liquid, or said gas, said conductive tip electrode positioned between 0 and 200 µm of said free end of said cantilever, wherein said conductive tip electrode is electrically isolated from said heater-thermometer;
- d. a first doped single crystal silicon electrode electrically connected to said heater-thermometer;
- e. a second electrode electrically connected to said heaterthermometer and said conductive tip electrode;
- g. a layer of insulating material positioned to provide electrical isolation between said conductive tip electrode and said heater-thermometer;

wherein one or both of said first doped single crystal silicon electrode and said second electrode comprise one or more legs of said cantilever.

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