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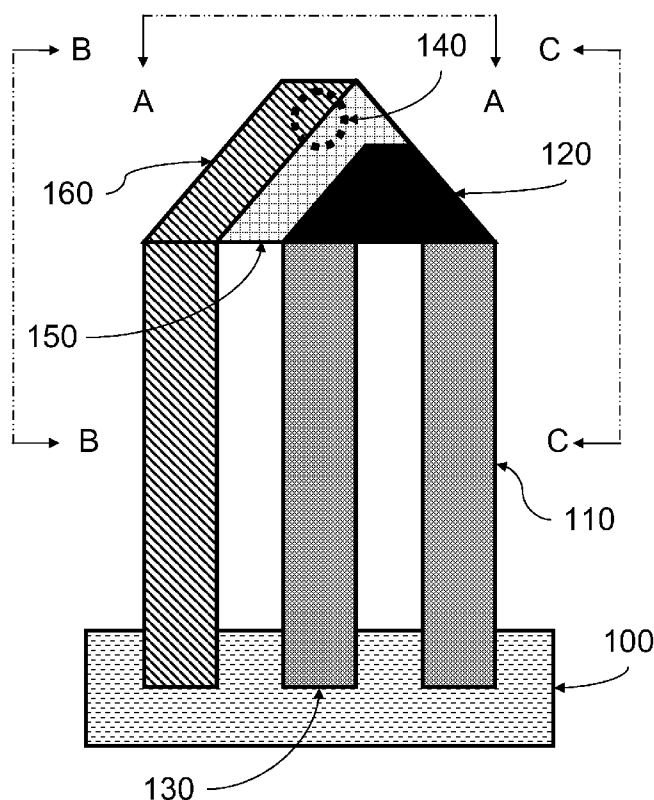


Figure 2

(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.

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TEMPERATURE-DEPENDENT NANOSCALE CONTACT POTENTIAL**MEASUREMENT TECHNIQUE AND DEVICE****CROSS REFERENCE TO RELATED APPLICATION**

[0001] This application claims the benefit of and priority to United States Provisional Application 61/024,962, filed on January 31, 2008, which is hereby incorporated by reference in its entirety.

BACKGROUND OF THE INVENTION

[0002] 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 actuating temperature-dependant electrical potential with nanometer scale resolution.

[0003] Microcantilever devices having integrated resistive heaters have found use in the fields of microscopy and information storage. For example, U.S. Patent Nos. 6,762,402 and 7,038,996 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 No. US2006/0238206.

[0004] 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 electrically isolated from the resistive heater which allows for temperature dependent Kelvin probe microscopy measurements as well as other uses.

SUMMARY OF THE INVENTION

[0005] 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 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 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.

[0006] In an embodiment, a microcantilever device of the present 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 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 heater-thermometer or conductive tip between 0 and 200 μm of the cantilever free end, preferably for some applications between 0 and 50 μm or between 0 and 25 μm of the free end of the cantilever. This expression also includes embodiments where 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.

[0007] 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.

[0008] In another embodiment, a microcantilever of the present 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.

[0009] In another aspect, a microcantilever is capable of producing and/or produces a change in the electrical potential of a 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.

[0010] 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 heater-thermometer. 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.

[0011] In another exemplary embodiment, a microcantilever is capable of providing and/or provides nanometer resolution 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 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 piezoelectric sensors within the cantilever, measurement of tunneling current, or any combination of these or other methods useful for sensing the surface profile.

[0012] In another aspect, provided is a method of sensing an attribute of a surface, the method comprising the steps of: 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 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 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 embodiment, the electrical potential of the surface is sensed by measuring a voltage or electric potential of a conductive tip of the microcantilever device.

[0013] In another aspect, the present invention provides a method of controlling an attribute of a surface, the method comprising 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.

[0014] Surface attributes useful for controlling with the methods described herein comprise the temperature of the surface, the electric 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.

[0015] 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 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 thermal interaction with the heater-thermometer portion of the microcantilever device and providing a second current 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.

[0016] 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.

[0017] 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 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 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 heater-thermometer.

[0018] 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.

[0019] 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.

[0020] 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

[0021] Figure 1 provides example data showing that the tip temperature can be calibrated as a function of the electrical resistance.

[0022] Figure 2 provides a schematic showing an overhead view of a first embodiment of a microcantilever device.

[0023] Figure 3 provides a schematic showing a front view of a first embodiment of a microcantilever device.

[0024] Figure 4 provides a schematic showing a side view of a first embodiment of a microcantilever device.

[0025] Figure 5 provides a schematic showing a side view of a first embodiment of a microcantilever device.

[0026] Figure 6 provides a schematic showing an overhead view of a second embodiment of a microcantilever device.

[0027] Figure 7 provides a schematic showing a rear view of a second embodiment of a microcantilever device.

[0028] Figure 8 provides a schematic showing a side view of a second embodiment of a microcantilever device.

[0029] Figure 9 provides perspectives views of a second embodiment of a microcantilever device.

[0030] Figure 10 provides a schematic showing an overhead view of a third embodiment of a microcantilever device.

[0031] Figure 11 provides a schematic showing a rear view of a third embodiment of a microcantilever device.

[0032] Figure 12 provides a schematic showing a side view of a third embodiment of a microcantilever device.

[0033] Figure 13 provides perspective views of a third embodiment of a microcantilever device.

DETAILED DESCRIPTION OF THE INVENTION

[0034] 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.

[0035] “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 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 a phosphorus concentration of about $1 \times 10^{17} \text{ cm}^{-3}$. A heater-thermometer can refer to a single or separate distinct elements for measuring and actuating the temperature, for example a thermistor or a thermocouple and a resistive heater.

[0036] “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.

[0037] “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.

[0038] “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.

[0039] “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 electrically 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.

[0040] “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 . 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.

[0041] 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.

[0042] In an embodiment, a device of the present invention comprises a cantilever having a fixed end and a free end. Useful 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 silicon. 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 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 legs.

[0043] 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 useful 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, 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 semiconductor 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 semiconductor having higher resistances can be useful as resistive heaters or thermistors or heater-thermometers.

[0044] 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 this way, the heater-thermometer can be resistively heated. Useful heater-thermometers are also capable of determining 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. Figure 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

[0045] 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 dip-pen 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 tip.

[0046] 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 heater-thermometer; 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 heater-thermometer may be adjacent to or on top of one another.

[0047] Referring now to the drawings, Figure 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**.

[0048] Figure 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**. Figure 4 shows a view of the first preferred embodiment in the B—B direction, showing conductive tip **140** and third electrode **160**. Figure 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.

[0049] Figure 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**. Figure 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**. Figure 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**. Figure 9 shows perspective views of an additional interpretation of the second preferred embodiment.

[0050] Figure 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**. Figure 11 shows a cross sectional view of the third preferred embodiment in the F—F direction and Figure 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**. Figure 13 shows perspective views of an additional interpretation of the third preferred embodiment.

[0051] 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.

[0052] 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

[0053] The AFM cantilevers of the present invention comprise an integrated resistive heating element and an electrically-addressable 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.

[0054] Figure 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.

[0055] 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 $2 \times 10^{14} \text{ cm}^{-3}$ having a resistivity of approximately 4 $\Omega\text{-cm}$. 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 $1 \times 10^{20} \text{ cm}^{-3}$ and the heater region near the free end of the cantilever is doped to $1 \times 10^{17} \text{ cm}^{-3}$. 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.

[0056] 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.

[0057] The cantilever temperature can be calibrated using infrared (IR) and Raman microscopy. Figure 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.

[0058] In the cantilever schematic of Figure 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 Figures 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.

REFERENCES

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[0062] J. Lee, T. L. Wright, T. W. Beecham, S. Graham, and W. P. King, "Electrical, Thermal, and Mechanical Characterization of Heated Microcantilevers," *Journal of Microelectromechanical Systems*, vol. In Press, 2006.

[0063] U.S. Patent 6,762,402.

[0064] U.S. Patent 7,038,996.

[0065] U.S. Patent Application Publication No. US2006/0238206.

STATEMENTS REGARDING INCORPORATION BY REFERENCE AND VARIATIONS

[0066] 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).

[0067] 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.

[0068] 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 group and all combinations and subcombinations possible of the group are intended to be individually included in the disclosure.

[0069] 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 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 art-known 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.

[0070] 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.

[0071] 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 device comprising:
 - a. a cantilever having a fixed end and a free end;
 - b. a heater-thermometer positioned near said free end of said cantilever; and
 - c. a conductive tip positioned near said free end of said cantilever, wherein said conductive tip is electrically isolated from said heater-thermometer.
2. The device of claim 1 further comprising one or more electrodes electrically connected to said heater-thermometer or said conductive tip.
3. The device of claim 2 wherein said one or more electrodes comprise a first electrode electrically connected to said heater-thermometer, a second electrode electrically connected to said heater-thermometer, and a third electrode electrically connected to said conductive tip.
4. The device of claim 2 wherein said one or more electrodes comprise a first electrode electrically connected to said heater-thermometer, and a second electrode electrically connected to both said heater-thermometer and said conductive tip.
5. The device of claim 2 wherein said one or more electrodes comprise doped semiconductor.
6. The device of claim 5 wherein said doped semiconductor is selected from the group consisting of doped diamond and doped silicon.
7. The device of claim 2 wherein said one or more electrodes comprise one or more legs of said cantilever.

8. The device of claim 2 wherein said one or more electrodes deliver electrical current to said heater-thermometer for effecting a temperature change in said heater-thermometer.
9. The device of claim 2 wherein said one or more electrodes provide a voltage or current to or from said conductive tip.
10. The device of claim 2 wherein said one or more electrodes comprise a material that can withstand a temperature up to 1250°C.
11. The device of claim 2 wherein said one or more electrodes comprise a metal.
12. The device of claim 11 wherein said metal is selected from the group consisting of tungsten, gold, aluminum, platinum, and nickel.
13. The device of claim 1 wherein said heater-thermometer comprises a thermistor.
14. The device of claim 1 wherein said heater-thermometer comprises a doped semiconductor.
15. The device of claim 1 wherein said heater-thermometer comprises a material that can withstand a temperature up to 1250°C.
16. The device of claim 1 wherein said conductive tip comprises a metal or a doped semiconductor.
17. The device of claim 16 wherein said metal is selected from the group consisting of tungsten, gold, aluminum, platinum, and nickel.
18. The device of claim 16 wherein said doped semiconductor is selected from the group consisting of doped diamond and doped silicon.
19. The device of claim 1 further comprising a layer of insulating material selected from the group consisting of undoped silicon, silicon oxide, silicon nitride, diamond, and polymer, wherein said layer of insulating material is positioned to electrically isolate said conductive tip from said heater-thermometer.

20. The device of claim 1 wherein said electrical isolation also provides thermal insulation between the conductive tip and said heater-thermometer.
21. The device of claim 1 wherein said electrical isolation allows for thermal communication between the conductive tip and said heater-thermometer.
22. The device of claim 1 wherein said heater-thermometer is spatially offset from said conductive tip.
23. The device of claim 1 wherein said heater-thermometer is located on top of said conductive tip.
24. 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 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;
 - ii. a heater-thermometer positioned near said free end of said cantilever; and
 - iii. said conductive tip positioned near said free end of said cantilever, wherein said conductive tip is electrically isolated from said heater-thermometer;
 - c. measuring an electrical property of said heater-thermometer, said conductive tip, or both, thereby sensing said attribute of said surface, said liquid, or said gas.
25. The method of claim 24 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.

26. The method of claim 24 wherein said temperature of said surface, said liquid, or said gas is sensed by measuring a resistance across said heater-thermometer.
27. The method of claim 24 wherein said electrical potential of said surface, said liquid, or said gas is sensed by measuring a voltage of said conductive tip.
28. The method of claim 24 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.
29. 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 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;
 - ii. a heater-thermometer positioned near said free end of said cantilever; and
 - iii. said conductive tip positioned near said free end of said cantilever, wherein said conductive tip is electrically isolated from said heater-thermometer; and
 - c. providing a voltage or current to said heater-thermometer, said conductive tip, or both.
30. The method of claim 29 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.

31. The method of claim 29 wherein said temperature of said surface, said liquid, or said gas is controlled by providing a current to said heater-thermometer.

32. The method of claim 29 wherein said electrical potential of said surface, said liquid, or said gas is controlled by providing a voltage to said conductive tip.

33. A method of manipulating a surface, said method comprising:

- a. providing a surface;
- b. providing a device having a conductive tip in thermal or electrical communication with said surface, said device comprising:
 - i. a cantilever having a fixed end and a free end;
 - ii. a heater-thermometer positioned near said free end of said cantilever; and
 - iii. said conductive tip positioned near said free end of said cantilever, wherein said conductive tip is electrically isolated from said heater-thermometer; and
- c. providing a voltage or current to said heater-thermometer, said conductive tip, or both.

34. The method of claim 33 wherein said manipulation of 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 electrons into said surface; and depositing material onto said surface.

35. The method of claim 33 further comprising

- d. allowing said surface to have thermal interaction with said heater-thermometer; and

- e. providing a second voltage or current to said heater-thermometer, said conductive tip, or both.

36. The method of claim 35 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 electrons into said surface; and depositing material onto said surface.

37. A method of manipulating a surface, said method comprising:

- a. providing a surface;
- b. providing a device having a conductive tip in thermal or electrical communication with a gas or a liquid between said surface and said conductive tip, said device comprising:
 - i. a cantilever having a fixed end and a free end;
 - ii. a heater-thermometer positioned near said free end of said cantilever; and
 - iii. said conductive tip positioned near said free end of said cantilever, wherein said conductive tip is electrically isolated from said heater-thermometer; and
- c. providing a voltage or current to said heater-thermometer, said conductive tip, or both.

38. The method of claim 37 wherein the current or voltage provided in step c results in a temperature change of said heater-thermometer and said liquid or said gas, or a discharge from said conductive tip to said liquid or said gas.

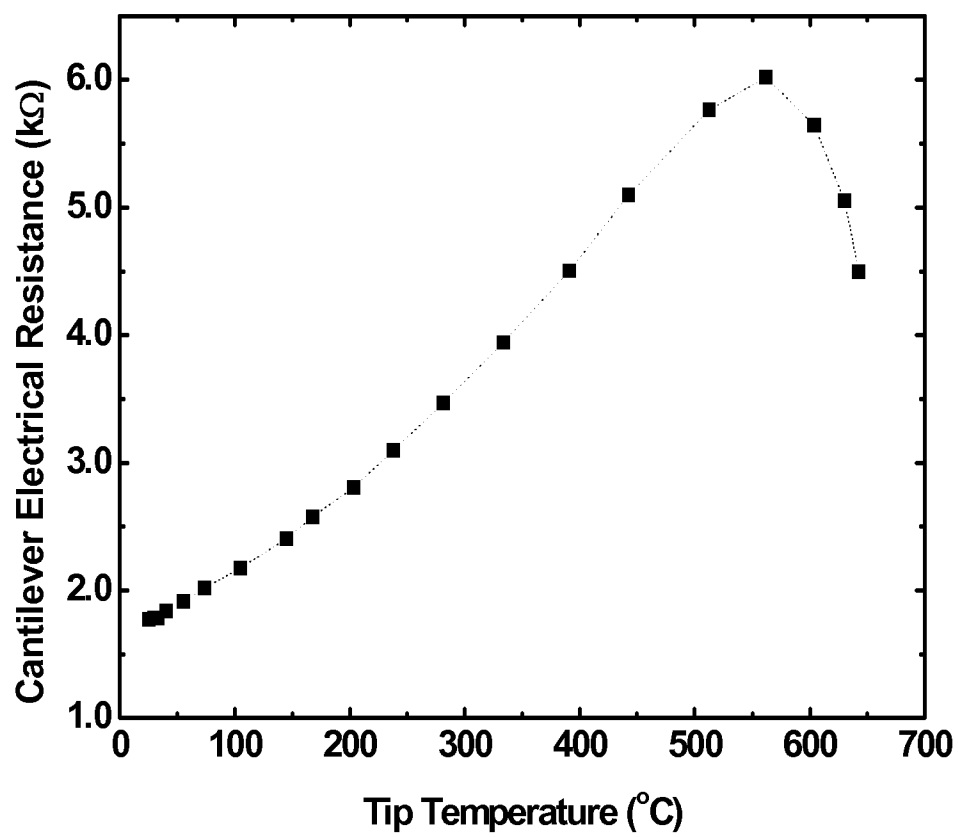
39. The method of claim 38 wherein said temperature change of said liquid or said gas or said discharge from said conductive tip to said liquid or said gas results in a chemical or physical reaction of said surface with said liquid or said gas.

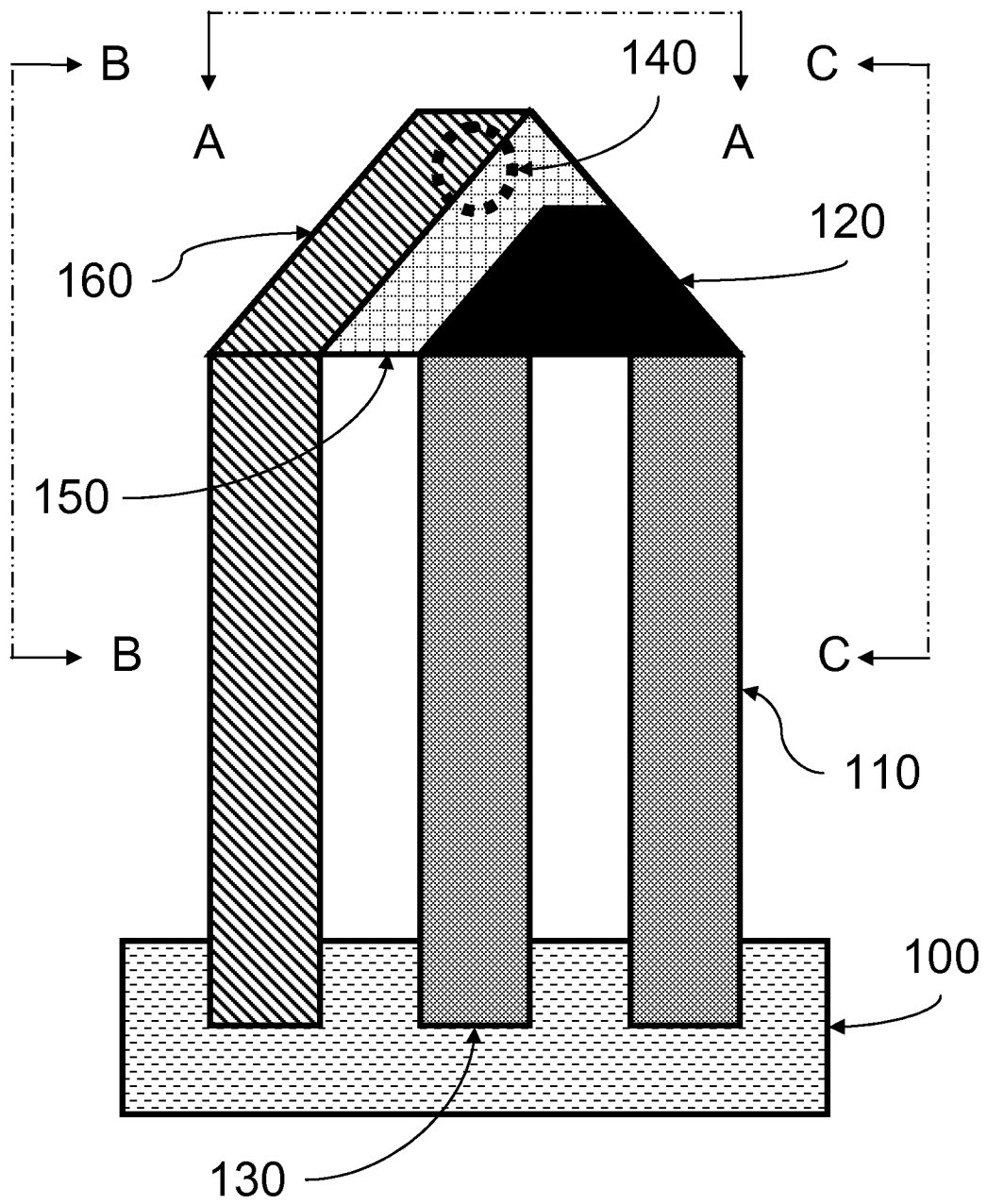
40. A device comprising:

- a. a cantilever having a fixed end and a free end;
- b. a heater-thermometer positioned between 0 and 200 μm of said free end of said cantilever; and
- c. a conductive tip positioned between 0 and 200 μm of said free end of said cantilever, wherein said conductive tip is electrically isolated from said heater-thermometer.
- d. a first electrode electrically connected to said heater-thermometer;
- e. a second electrode electrically connected to said heater-thermometer; and
- f. a third electrode electrically connected to said conductive tip.

41. A device comprising:

- a. a cantilever having a fixed end and a free end;
- b. a heater-thermometer positioned between 0 and 200 μm of said free end of said cantilever; and
- c. a conductive tip positioned between 0 and 200 μm of said free end of said cantilever, wherein said conductive tip is electrically isolated from said heater-thermometer.
- d. a first electrode electrically connected to said heater-thermometer; and
- e. a second electrode electrically connected to said heater-thermometer and said conductive tip.

**Figure 1**

**Figure 2**

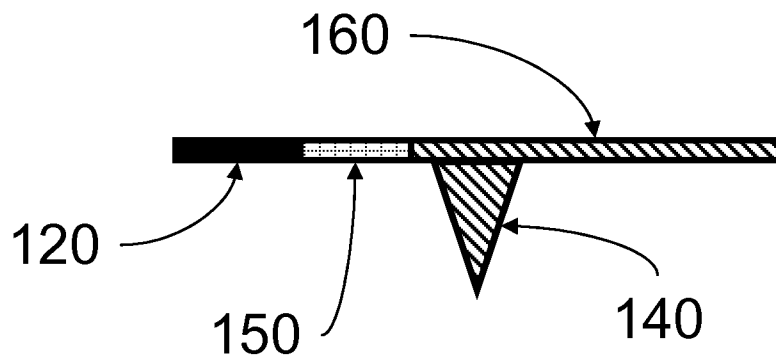


Figure 3

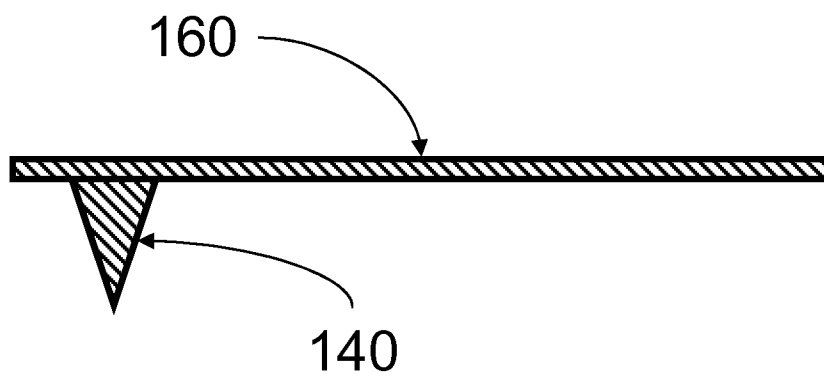


Figure 4

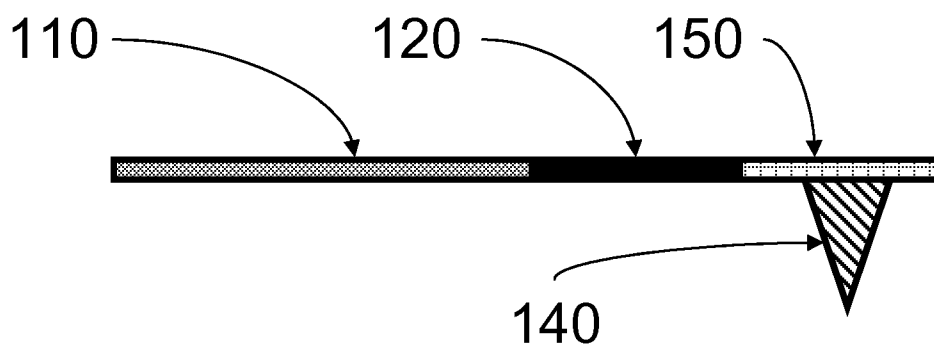


Figure 5

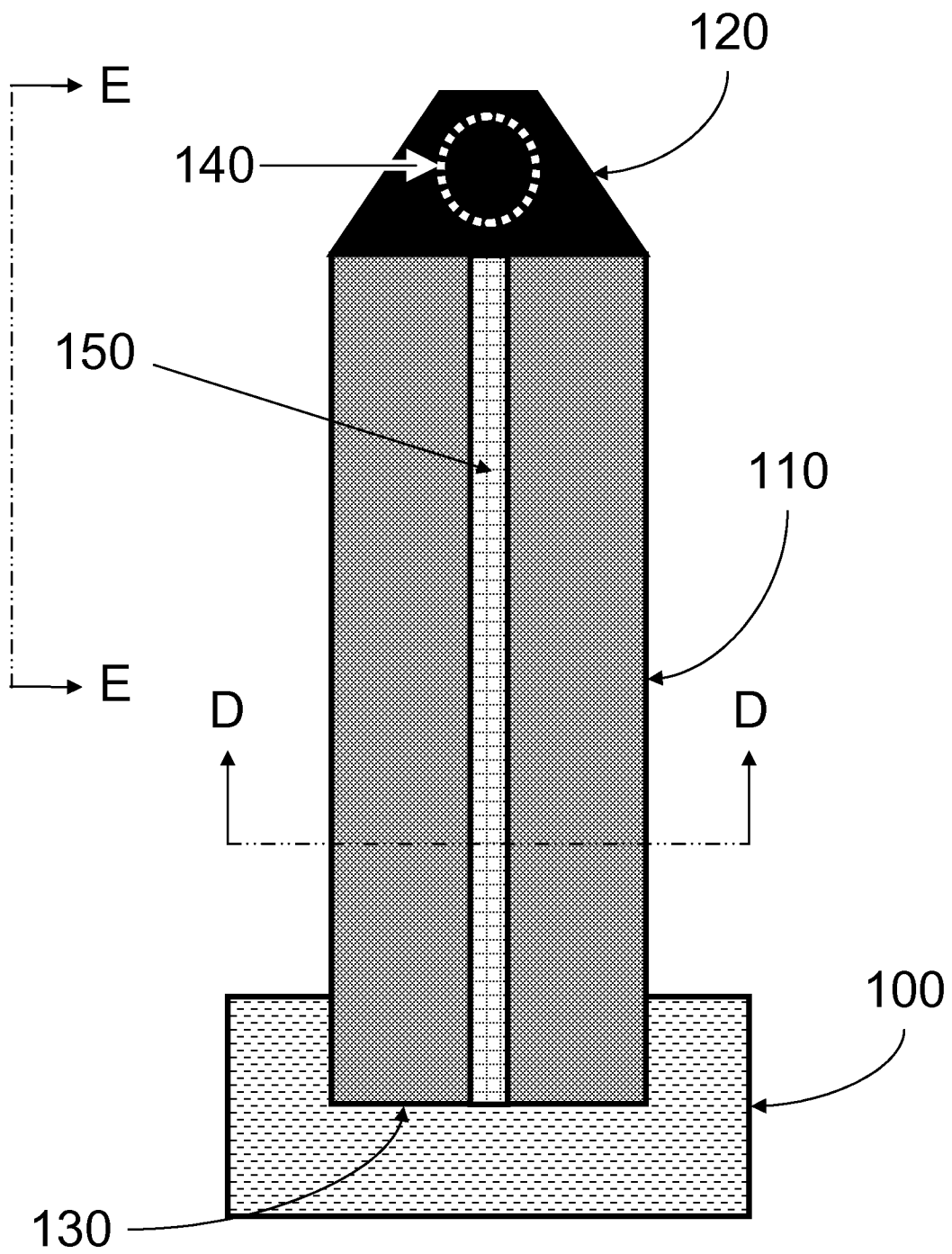


Figure 6

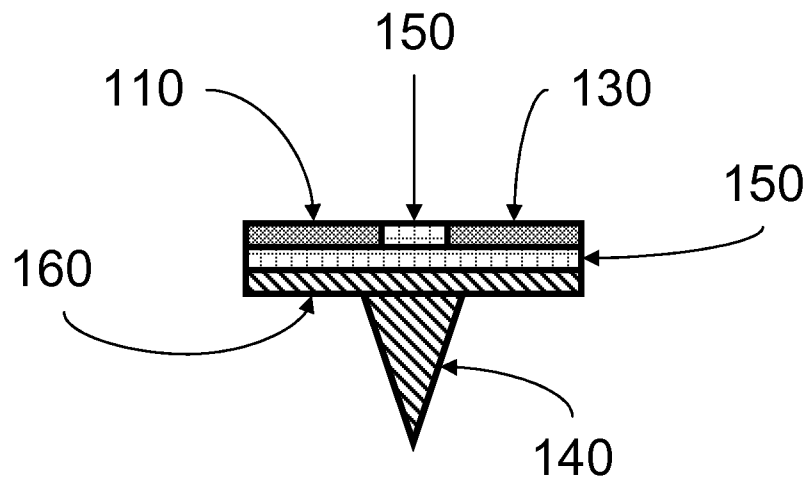


Figure 7

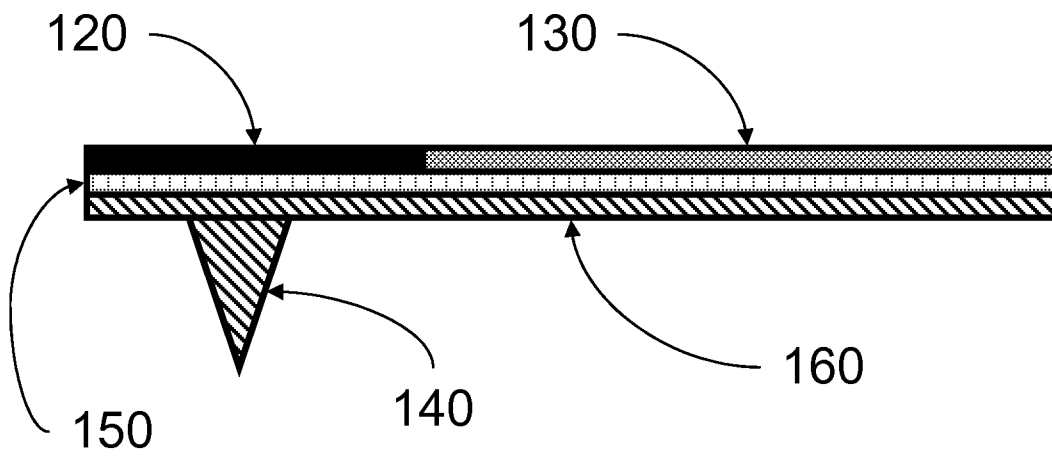


Figure 8

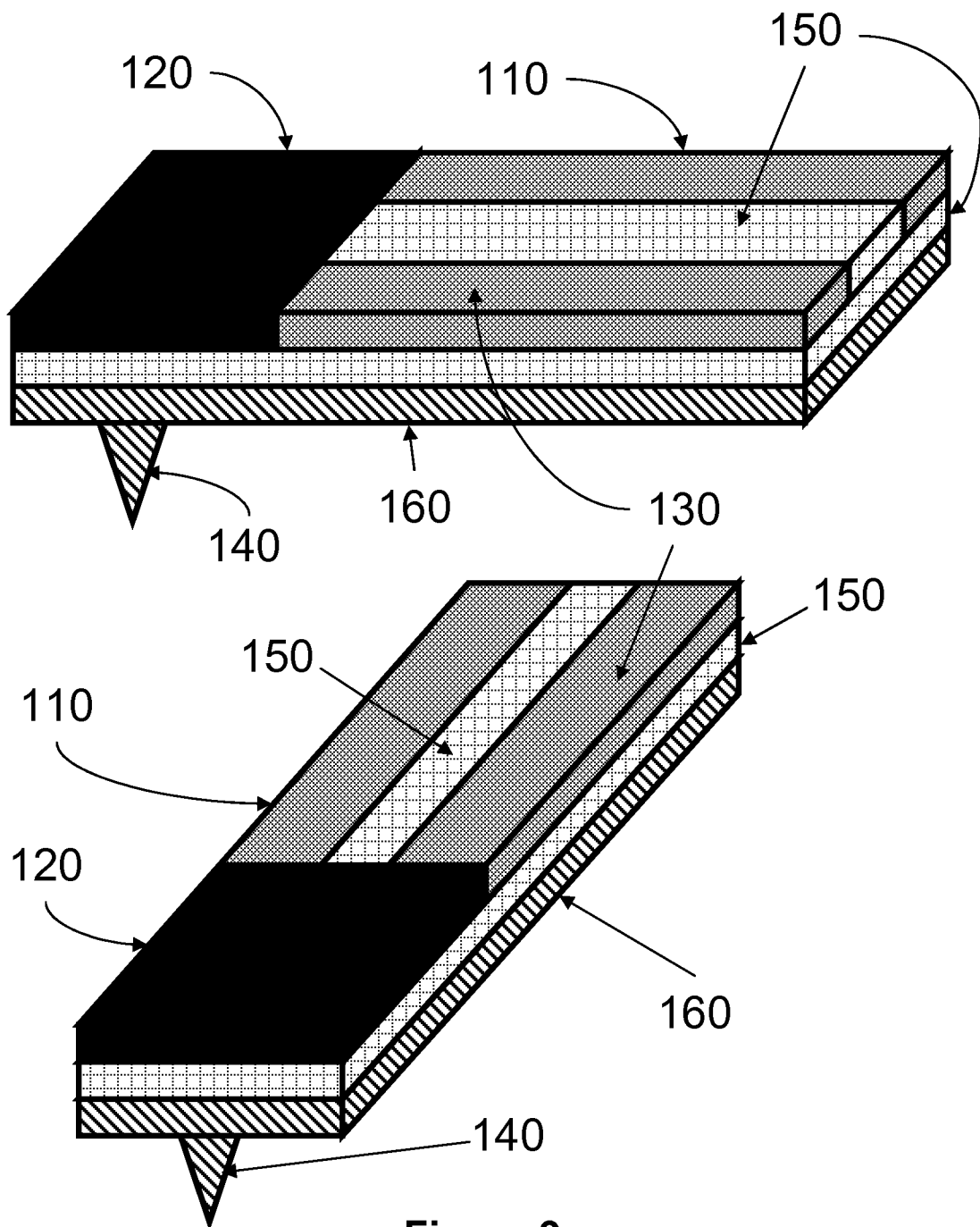
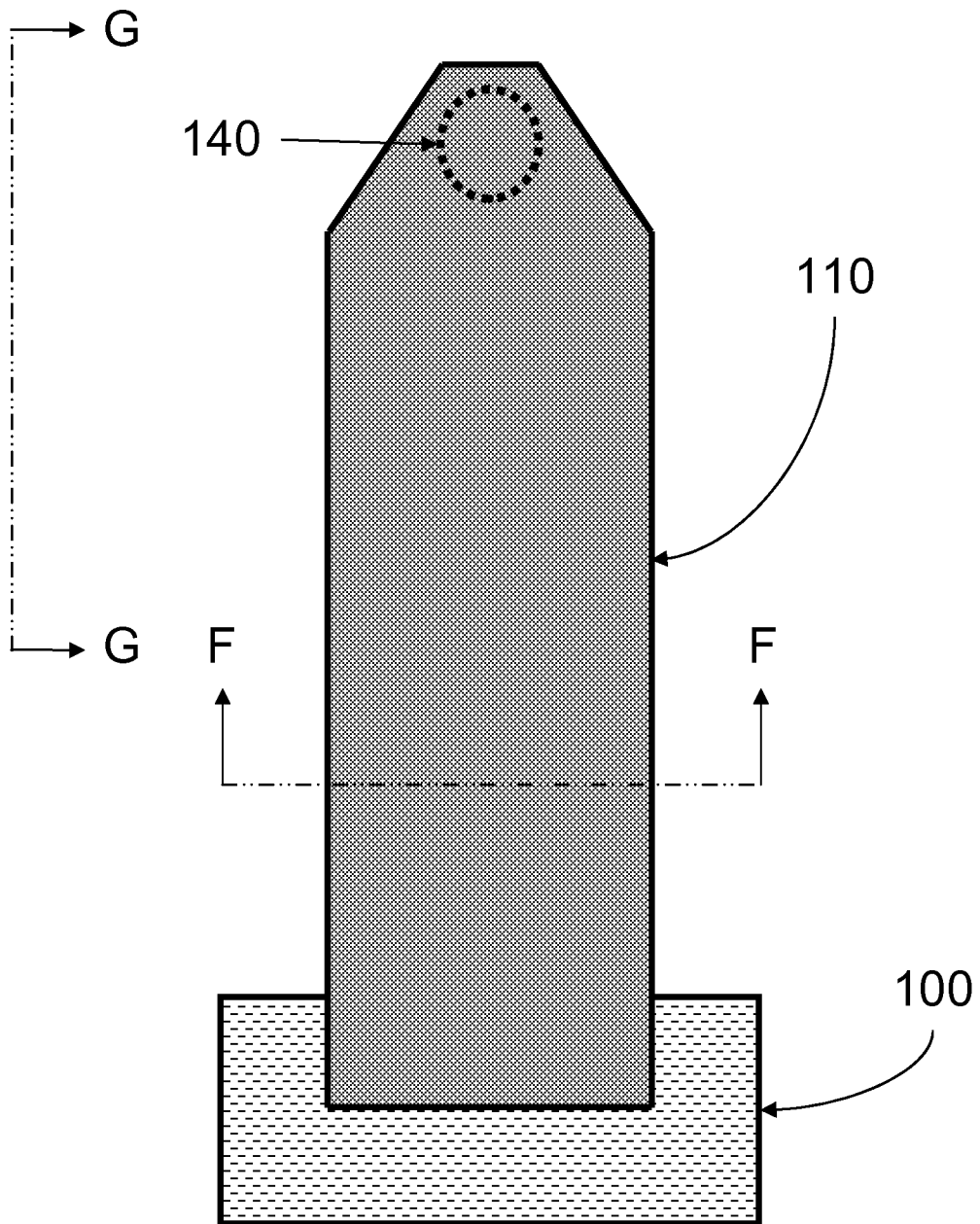


Figure 9

**Figure 10**

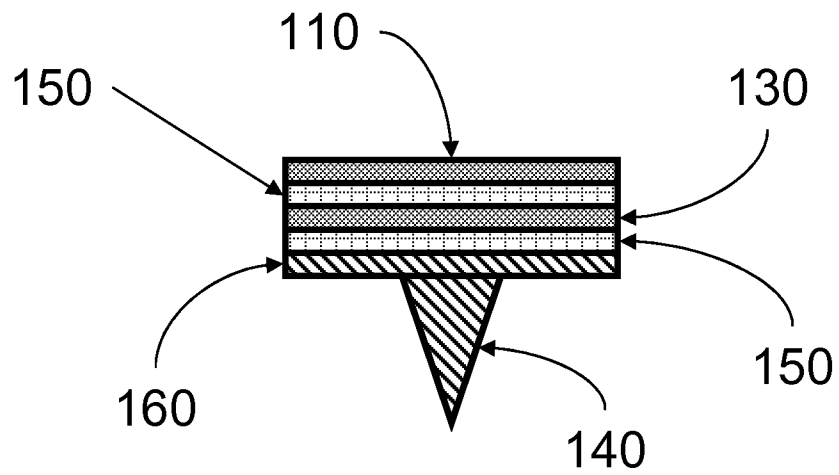


Figure 11

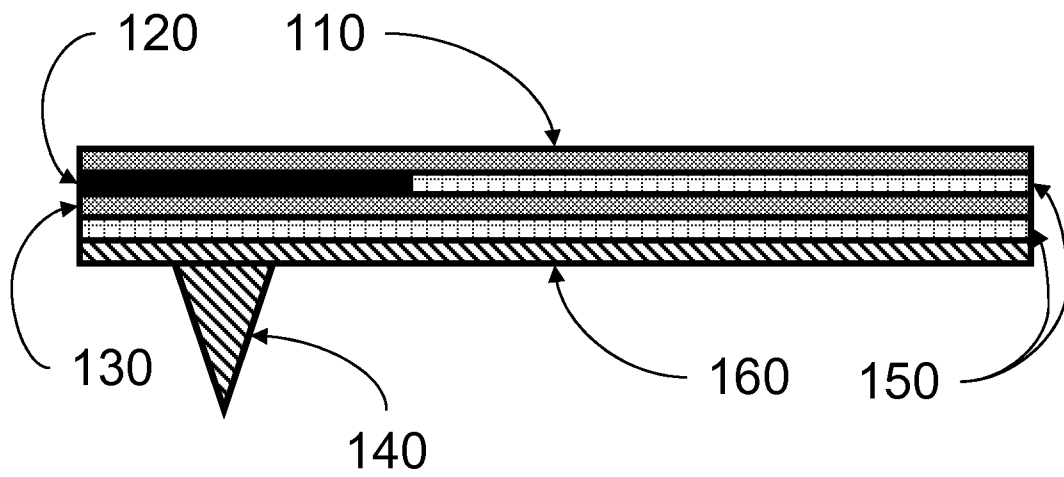


Figure 12

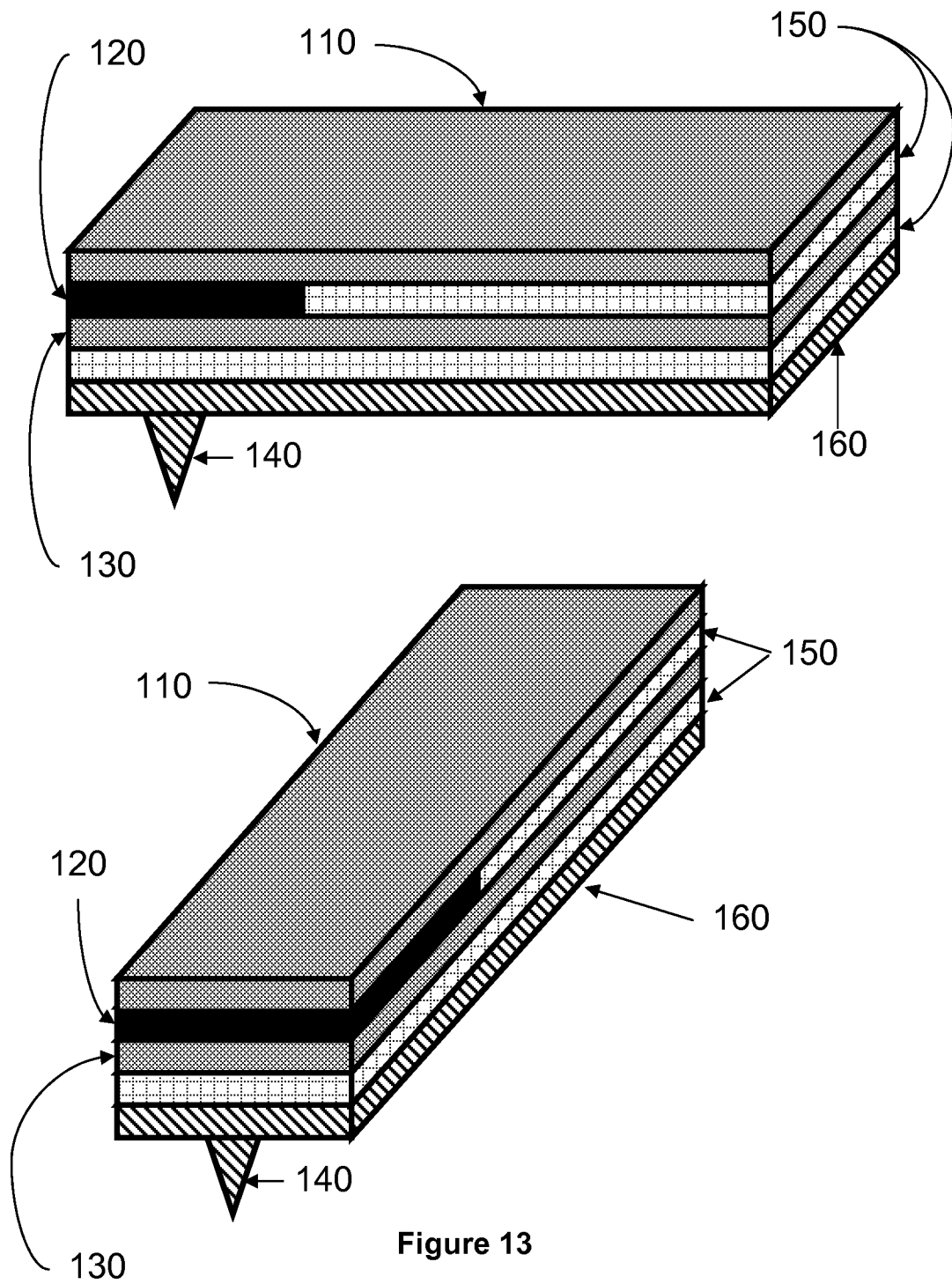


Figure 13

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 09/32545

A. CLASSIFICATION OF SUBJECT MATTER

IPC(8) - G01R 31/00 (2009.01)

USPC - 324/750; 977/732

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC(8) - G01R 31/00 (2009.01)

USPC - 324/750; 977/732

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
USPC - 324/750, 754, 758; 977/732, 724

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

PubWEST(PGPB,USPT,USOC,EPAB,JPAB); WIPO; Web: Google, Google Scholar

Search Terms Used: cantilever, heater-thermometer, conductive tip, electrode, doped semiconductor, diamond, silicon, current, temperature change, voltage, metal, sensing, surface temperature, electrical potential, resistance

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ----- Y	US 2006/0254345 A1 (King et al.) 16 November 2006 (16.11.2006) Entire document, especially: abstract, Fig 6-8; para [0007]-[0011], [0022]-[0025], [0040]	1, 2, 7, 14, 16, 18, 20-23 ----- 3-6, 8-13, 15, 17, 19, 40, 41
X	Kim, K. J., Park, K., Lee, J., Zhang, Z.M., King, W.P., "Nanotopographical Imaging using a Heated Atomic Force Microscope Cantilever Probe," 2007, Sensors and Actuators A, Vol. 136, December 2006, p 95-103. (abstract) [online]. Retrieved from the Internet on [16 March 2009]. Retrieved from: <URL: http://www.sciencedirect.com/science?_ob=MIimg&_imagekey=B6THG-4MH2BV1-1-K&_cdi=5282&_user=952835&_orig=search&_coverDate=05%2F01%2F2007&_sk=998639998&view=c&wchp=dGLbVtz-zSkzk&md5=30fd69aacb9ed8fe6a3ee9b85b797d0c&ie=/sdarticle.pdf > Entire document, especially: abstract; Fig 1, 2, 3, 4, 6b, 6c, 6d; abstract, p 96, 99-100	24-28
X ----- Y	Remmert, J.L., Yan, W., Lee, J., Shannon, M.A., King, W.P., "Contact potential measurement using a heated atomic force microscope tip," 2007, Applied Physics Letters, Vol. 91, Issue 14, October 2007, 143111 p. 1-3. (abstract) [online]. Retrieved from the Internet on [16 March 2009]. Retrieved from: <URL: http://mechse.illinois.edu/research/shannon/publications/pdf/2007APLJLR.pdf > Entire document, especially: Fig 1a, 3a, 3b; p 1, 2	29-32 ----- 3, 4, 9, 11, 12, 17, 40, 41

☒ Further documents are listed in the continuation of Box C.

* Special categories of cited documents:

"A" document defining the general state of the art which is not considered to be of particular relevance

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search

18 March 2009 (18.03.2009)

Date of mailing of the international search report

09 APR 2009

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Authorized officer:

Lee W. Young

PCT Helpdesk: 571-272-4300

PCT OSP: 571-272-7774

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 09/32545

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2007/0114401 A1 (King et al.) 24 May 2007 (24.05.2007)	33-36
Y	Entire document, especially: Fig 1A-C, 3A, 4; para [0031], [0034], [0035], [0037], [0038], [0041], [0042]	37-39
Y	Lee J., "Fabrication, characterization, and application of multifunctional microcantilever heaters," 2007, Ph.D. Dissertation, Georgia Institute of Technology, May 2007. (abstract) [online]. Retrieved from the Internet on [16 March 2009]. Retrieved from: <URL: http://etd.gatech.edu/theses/available/etd-04022007-144122/unrestricted/lee_jungchul_200705_phd.pdf > Entire document, especially: Fig 2.2, 2.3a, 2.3b, 2.4a, 2.4b; p 20, 21, 23-25, 54; table 3.1	5, 6, 8, 10, 13
Y	Nelson, B.A., "Nanoscale thermal processing using a heated atomic force microscope tip," 2007, Ph.D. Dissertation, Georgia Institute of Technology, May 2007. (abstract) [online]. Retrieved from the Internet on [16 March 2009]. Retrieved from: <URL: http://smartech.gatech.edu/bitstream/1853/14512/1/nelson_brent_a_200705_phd.pdf > Entire document, especially: p 14, 15, 18	15, 19
Y	Lee, J., Naeli, K., Hunter, H., Berg, J., Wright, T., Courcimault, C., Naik, N., Allen, M., Brand, O., Glezer, A., King, W.P., "Characterization of Liquid and Gaseous Micro-and Nanojets using Microcantilever Sensors," 2007, Sensors and Actuators A, Vol. 134, p 128-139, June 2006. (abstract) [online]. Retrieved from the Internet on [16 March 2009]. Retrieved from: <URL: http://www.mems.gatech.edu/msmaweb/site_2006/publications/publication_list_files/2007/Characterization%20of%20liquid%20and%20gaseous%20micro-%20and%20nanojets%20using%20microcantilever%20sensors.pdf > Entire document, especially: Fig 4; p 129, 131, 133	37-39