Title: SENSORS FOR SCANNING PROBE MICROSCOPY, METHOD FOR THREE-DIMENSIONAL MEASUREMENT AND METHOD FOR MANUFACTURING SUCH SENSORS

Abstract: The sensors for SPM consist of a body, microcantilever and probe member, having a common flat surface in which at least one functionalization element shaped as trench and/or opening is formed with a heterogeneous probe element assembled in it, such as carbon nanotube (CNT) or other type nano-sized tubes, fibers, micro-crystals etc., including such with a complex shape and specially functionalized. In a sensor embodiment piezoresistors are used for transducing the bending oscillation of the microcantilever and probe member in electrical signal. The three-dimensional measurement method allows using common scanning microscopy system, in a particular point of the scanning grid to perform measurement in all three directions without translating/rotating the system and/or the sample or change the sensor, by controlled periodic actuation of sensor with microcantilever and probe member with individual oscillation characteristics of bending without torsion in each direction of measurement, which characteristics are discernible from one another upon measurement and the number of the probe elements used is sufficient to ensure measurement in each of the three directions. The invention includes also a method for manufacturing the described sensor.
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GW, ML, MR, NE, SN, TD, TG).

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TECHNICAL FIELD

This invention is related to sensors for scanning probe microscopy (SPM) providing high measurement accuracy and resolution, a method for three-dimensional measurement with feedback control with such sensors and a method for their manufacturing. These sensors through detection of mechanical oscillation parameters like perturbations in phase, amplitude magnitude, resonance frequency or tunneling current are used to determine the surface morphology or composition, geometrical size or other sample characteristics in various technical areas. More particularly, the invention relates to sensors, in which heterogeneous probe elements are used, a method for three-dimensional measurement and a method for the manufacturing of such sensors, which are to be implemented in atomic force microscopy (AFM) and scanning tunneling microscopy (STM) systems.

BACKGROUND ART

The scanning probe microscopy (SPM) and its most used forms - AFM and STM are widely used contemporary methods for advanced high resolution surface analysis, research in the field of nano-sized objects, micro/nano manipulations, etc. Data acquisition for SPM is accomplished by detecting the interaction between a sensor and a sample within a grid of pre-determined by the scanning system points and usually SPM methods employ sensors containing microcantilevers or some other type of micromechanical flexible elements with sharp edges or probe elements (also named probes). Microcantilevers react to small influences and transduce an influence caused by an investigated sample into a measurable physical quantity as they bend and/or a perturbation of the mechanical oscillation or tunneling current from the sample investigated is detected. An example of such commonly used sensor is shown on Fig.1. This sensor has a body 1 out of which a microcantilever 2 with length \( L \), width \( W \) and thickness \( H \) is formed. The microcantilever has one free end with a corresponding
direction of preferable bending along the Z axis, while in its free end a probe member 3 with a probe element (probe) P is formed, which interacts particularly with the sample investigated. The free end of such microcantilever would be deflected under the action of the force $F$ directed along the Z axis to the distance $\Delta Z$ according to the relation

$$F = k \cdot \Delta Z$$

(1)

where $k$ is the stiffness coefficient, determined by geometrical size and material properties of the cantilever. For a homogeneous rectangular microcantilever in a number of sources, $k$ is described by the relation:

$$k = E \cdot W \cdot H^3/(4L^3)$$

(2)

where $E$ is the Young's modulus (for <110> silicon $E = 1.7 \cdot 10^{11}$ N/m²).

In addition the microcantilever has a resonance frequency $f_r$ of bending oscillation along the Z axis defined by the relation:

$$f_r = 0.162 \cdot (E/p)^{1/2} \cdot H \cdot L^{-2}$$

(3)

where $p$ denotes the density of the material (for silicon $p = 23 \cdot 10^3$ kg.m⁻³).

The interaction between probe element and sample is determined by the shape and composition of the probe element. As a result of the interaction the microcantilever bends in $\Delta Z$ and/or the magnitude of amplitude and phase of oscillation change. The change of these parameters is detected by different methods, such as optical, capacitive, piezoelectric, piezoresistive etc., which are well known to those, skilled in the art.

To obtain probe elements with different size and shape made of the material of the substrate a number of methods have been developed. Such as are disclosed in the patents US 5051379, US 5242541, US 5345815, US 5345816, US 561 1942.

Typical of the methods for manufacturing such sensors is that the probe element is produced by etching, which etching is carried out on a dominant part of the sensor surface, the process chain sequence is long because the probe elements are formed first, then the remaining micromechanical elements on the newly formed surface after etching are obtained. Furthermore after the probes structures are produced, the structures with convex elements are processed and this requires specialized equipment and materials in subsequent processing. In addition, as a consequence of the etching process employed, limitations to the form and parameters of the obtained sharp tips exist, said form is
determined by the geometry and orientation of the etching mask and by anisotropy and selectivity of the processes employed; probes obtained in a single etching process are oriented unidirectional and due to technological reasons probes with crystallographically nonequivalent orientation cannot be produced simultaneously. This leads to the practical impossibility to obtain sensors with more than one probe oriented in different crystallographic directions.

To avoid part of above described difficulties, microcantilevers placed entirely in the plane of the substrate surface have been developed. Such devices are disclosed in patents US 5729026 and US 5856672, using a microcantilever with an integral tip, which is produced by photolithography and a subsequent anisotropic wet etching without forming a structure outside the plane of the microcantilever. This microcantilever bends preferably in a direction perpendicular to the substrate surface while the placement of the integral tip within the plane of the substrate allows employing the sensor only at a relatively big angle to the surface of the sample.

The resolution of sensor for a SPM system depends on the shape and size of the probe element. To significantly improve said resolution in recent years a number of research studies on the integration of carbon nanotubes (CNTs) as probe elements were carried out. CNTs are the best known probe tips, because of their remarkable properties like an extreme aspect ratio; when are defect-free, they possess great mechanical strength, good electrical and thermal conductivity, their properties can be modified through functionalization, while they remain chemically stable. It is also known that under certain conditions CNTs with semi-spherical ends can be obtained.

A number of methods for the production of AFM sensors with CNTs are known, e.g. those described in patents US 6346189, US 6401526, US 6528785, US6756025, US 6871528, US 7048903, US 7138627 - in all of them previously formed tips of microcantilever material are employed, while the CNTs on top of the tips are produced mainly in two ways:
- high temperature or otherwise controlled CNTs synthesis - in a suitable environment on areas (isles) with catalytic features to the synthesis, the areas being placed on the tips;
- attachment of synthesized in advance CNTs on previously prepared tips.

Common deficiencies of both mentioned approaches for the production of AFM sensors with CNTs are the low throughput and reproducibility, due to the lack of methods for exact control of the growth, orientation and placement of the CNTs, as well as the necessity to employ complex and specialized equipment.

For example, patent application US 2008121029 discloses a method for obtaining AFM sensors with CNTs, consisting of a body, microcantilever extended from the body and a probe member formed in the vicinity of a free end of the microcantilever in the shape of a bent cone, pyramid or prism with a trench on it in the shape of a notch, which may as well be an opening. On the sidewall of the notch a CNT is attached in a manner jutting out from a terminal end portion of the probe member.

Specific for sensors obtained using this method is that the surfaces of the structures on which CNTs are fixed, form an angle with the plane of the surface of the substrate which is determined by the slope of the probe member shaped. In the patent application mentioned several alternative embodiments of the method are described which clearly state that the angles of the CNT towards the slope of the surface plane are of limited number and for each slope a specific and different technological approach is proposed. In this sense, the method of the patent mentioned does not disclose a general approach for the production of probes with probe elements placed under different angles, which in the first place is necessary to obtain optimum measurement results and a more effective production process as well.

The method for attaching the CNT to the sidewall of the notch presented in the above mentioned patent application requires the employment of specialized equipment like a scanning electron microscope, with the process of fixation being not sufficiently
precise to ensure repeatability of placement at the point of fixation, thus rendering the method difficult to employ in practice.

Patent application US 200801 1066 describes a method for producing a sensor for AFM, consisting of a sensor body, microcantilever shaped as a protruding member and a sharp tip (probe) entirely produced with a photolithographic patterning. To produce such a device, silicon-on-insulator (SOI) wafers are used, having described the opportunity of obtaining a varied form microcantilevers employing entirely photolithographic patterning. The existing lithographic methods provide for high precision and reproducibility of patterned elements in a wide range of sizes, obtained according to the patent application mentioned. Despite that, the method requires a special material for the substrate (SOI wafers) and a comparatively complex process sequence. In addition, in the step of forming a probe element with a tip, located in a plane different from that of the initial substrate surface plane via etching process, due to the presence of a connecting element in the pattern in practice it is impossible to achieve reproducible probe tip apex location relative to the remaining probe elements of the sensor.

In the cited patent application US 200801 1066, implementation of CNT for tips of AFM sensors is mentioned only as part of the background art, but which according to the inventors is a problem. This patent neither mentions nor presumes that it is possible to form and employ trenches and/or openings in the probe member, not to mention orienting them in a specific way as an element to be used for attaching of a CNT as a probe tip.

In the patent application WO 2009/000885 a method to obtain microcantilever sensors with a bimorph flat shaped structure is disclosed. The tips are shaped in a photolithographic process and etching strictly along the axis of the microcantilever while the microcantilever is bent through a bimorph thermo actuator and an additional heating element is used to control the length of the microcantilever. The preferred direction of microcantilever deflection is perpendicular to the surface of the substrate. According to the description, preferred sharp tips are CNTs attached to the end of the probe element. This patent application neither discloses any method for attaching these CNTs, which are
part of the claimed sensors by the application, nor is mentioned or presumed to use trenches and/or openings for such a purpose.

The sensors known to the present moment, which possess a single measurement direction, are employed in conventional SPM, where scanning systems with one or two surface scanning axes, orthogonal to the mentioned measurement direction are utilized. This type of systems are used to investigate samples, where the area of the surface to be scanned is determined in advance, as well as the direction to approach said area is known while the steepness of the profile of the elements within this surface area is restricted. When measuring of sample surface areas with complex shape or steep slope as well as the acquisition of two- and three-dimensional profiles is required, the samples are rotated relative to the measurement direction of the sensor in order to access these surface areas with known up to date sensors and scanning systems employed.

Patent application US 2008083270 discloses a system and a method for multidimensional detection and measurement of forces for the purpose of SPM with multidimensional sensitivity, in particular system and method employing CNT. Specific of this measurement method is that it employs the displacement of the apex of the probe element in various directions while different modes of cantilever oscillations are used - bending and torsion. To obtain a probe with CNT in controlled positions and controlled parameters, a catalytic synthesis on a prepared MEMS structure is used, as described in patent US 6146227 by the same inventor. In spite of the idea of a probe member with several probe elements, as illustrated in Fig. 20 in patent application US 2008083270, in this patent application as well as in patent US 6146227 no method whatever for the production of such MEMS with CNTs placed on different probe surfaces is disclosed - such a technical solution is missing in these documents. Moreover, as explained for Fig. 20 and Fig. 21 in patent application US 2008083270, an embodiment is disclosed where a CNT-oscillator with a cylindrical symmetry is realized, but it is not disclosed how the phase of oscillation of a probe mentioned, like the one depicted in Fig. 21, in more than one direction can be detected if no magnetic particle is attached at the probe end.
There is therefore a need for a microcantilever sensor with a probe element, providing high resolution, which sensor to perform high precision measurements under a desired angle towards the sample investigated. It is furthermore necessary to improve this sensor and extend its capabilities in a way to overcome the current impossibility to perform measurements in two and three dimensions in a single point of measurement with one and the same sensor. There is a need, moreover, for an adequate three-dimensional measurement method by which using such sensors with advanced features, fast and simple to make three-dimensional (and two-dimensional) accurate measurement with the SPM technique in any particular point of measurement without imposing additional rotation steps on the second direction or a new measurement for the third direction, in order to acquire all data in measured point and to obtain an overall profile or size of the investigated sample, opportunities which current measurement methods do not provide. Likewise there exists a need for development of a general, easy to be implemented and reproducible method, by which to produce the desired sensor with controlled mechanical oscillation characteristics, not only with a single, but with multiple probe elements, oriented under differing angles towards the axis of the microcantilever, in order to achieve practically the sought after improved capabilities and high sensor resolution and precision.

DISCLOSURE OF THE INVENTION

Definitions

Unless otherwise specified, relating to the description of the present invention and the accompanying claims certain definitions are implemented as follows:

The term "functionalization element", as it is used here, denotes a region of the probe portion, shaped concavely with a constant cross section, with the steepness of its walls and size of the functionalization element sufficient to accommodate a part of elongated heterogeneous probe element through positioning or self-positioning. The
functionalization element could be an opening or a trench. In both cases, after placing an elongated heterogeneous probe element within the functionalization element, the heterogeneous probe element is positioned or self-positioned in a selected in advance location. Functionalization elements are obtained through a patterned etching process in a single crystal silicon substrate, with or without additional sacrificial layers.

The term "composed trench" as it is used here, denotes any recess which results from at least two etching processes, the second of which covers only the bottom of said recess obtained during the first etching.

The term "heterogeneous probe element" (or "probe element" only), for the purpose of the present description and claims denotes an element used for the detection of a specific interaction, produced out of a material, different from the material of the substrate, for example carbon, boron, boron nitride, metals, zinc oxide etc., in the shape of a nanotube, nanowire, nanofibre, nanorod, micro/nanocrystal and similar. The heterogeneous probe element can be employed as it is obtained, or it may comprise of separately obtained heterogeneous parts, but it could previously be processed accordingly as to possess certain physical, chemical or other specific properties. In all cases, the heterogeneous probe element is capable of a specific interaction with the sample under investigation, resulting in a mechanical sensor response and/or an electrical signal.

The term "flexible micromechanical element" as used here, denotes a mechanical element sized in the micro- or nanometric range, which due to its shape and material it is built of, during the measurement transduces interaction with the investigated sample into mechanical response. In this description, depending on context, examples for flexible micromechanical elements are the microcantilever and probe member separately, as well as both together.

The term "flexible micromechanical structure" denotes a flexible micromechanical element in an intermediate state in the process of production, being already shaped on the common for the sensor of the present invention flat surface of the single crystal silicon.
substrate, but it is still non-released or its production process being still incomplete in some other sense.

The term "measurement direction" relates to the process of measurement employing excited oscillation of a flexible micromechanical element with a varying frequency in a frequency range including a resonance oscillation frequency of a flexible micromechanical element. A direction in which a flexible micromechanical element oscillates with resonance frequency is the direction of measurement. When the term "resonance frequency" is used in the present description, it is to be understood that it relates to the resonance oscillation of a flexible micromechanical element being in a bending mode without torsion along a certain direction. Unless explicitly otherwise noted, eigenfrequency usually is within the employed working frequency range.

The terms "test amplitude" and "working amplitude" refer to amplitudes differing in magnitude used in different regimes of the three-dimensional measurement according to the invention. The "test amplitude" is of a big magnitude, while the "working amplitude" is of a small magnitude.

The term "point of measurement" denotes a position of the probe element apex relative to the sample in which a measurement is performed. The "point of measurement" is part of a grid of points, characteristic for any scan system along which the sample is scanned in accordance of the system algorithm for tracking.

_Brief Description of the Invention_

In a first aspect, the present invention relates to a sensor for SPM, comprised of a body, a microcantilever protruding out of it and a probe member protruding at its free end, jointly shaped out of a single-crystal substrate and a probe element. Said sensor has a flat probe member, which is located in the same plane with the body and the microcantilever, forming their common flat surface. The probe member includes a probe portion with at least one concavely shaped functionalization element on it, where an elongated heterogeneous probe element is assembled, whose axis is parallel to the
common flat surface or is perpendicular to it. On the microcantilever and/or the probe member for each measurement direction $X$, $Y$ and/or $Z$ with said sensor, an area is individualized with determined geometric dimensions, which dimensions define the individual bending oscillation characteristics of said microcantilever and/or probe member in the relevant measurement direction and are determined in accordance with relations for discemibleness of the resonance frequencies, which are a preferred part of this aspect of the invention.

In other embodiments, the invention discloses SPM sensors with more than one probe functionalization element and accordingly assembled within them probe elements, with appropriately determined geometrical dimensions of the flexible micromechanical members of the sensor, thus permitting measurements in more than one direction, as is described in details further in this specification.

In accordance with the invention, said SPM sensors use elongated heterogeneous probe elements, selected among nanotubes, nanowire, nanofibres, nanorods, made of carbon, boron, boron nitride, metals, or micro-/nano-crystals like zinc oxide, preferably single nanotubes or a bundle of nanotubes, each of them selected out of a single- or multiwall carbon nanotubes, boron or boron nitride nanotubes. Probe elements can be constituted of heterogeneous parts with spherical, pyramidal or multi-pyramidal configuration or they can be additionally functionalized for specific applications, as well as be manufactured with a modified electrical conductivity.

In a preferred embodiment of the invention, piezoresistors transducing the displacement of the microcantilever and/or probe member in any measurement direction in an electrical signal are used.

In a second aspect, the invention discloses a method for three-dimensional measurements with feedback control with a sensor for SPM of the surface of a sample, where the sensor and/or the investigated sample are moved relative to each other by the scanning system to reach any one of the points on the surface where a measurement is carried out, in a manner sufficiently close to the surface to permit a measurement. In the method a sensor is used with a single microcantilever and a probe member with a
common flat surface and individual oscillation characteristics of bending without torsion in each direction of measurement, which characteristics are discernible from one another upon measurement. The sensor used has probe elements, whose number is sufficient to ensure measurement with feedback control in each of the directions X, Y and Z. When approaching the corresponding point on the sample surface, where measurement is carried out, the sensor and the sample are set in a static position and the surface surrounding of the probe element apex is subjected to investigation stepwisely in each of the directions X, Y and Z without translating the sensor and/or sample. The measurement with feedback control is performed through bringing the microcantilever and probe member into exited oscillation by periodic actuation, which oscillation is sequentially carried out with test amplitude magnitude and working amplitude magnitude for each of the directions X, Y and Z, and with a frequency in range containing the resonance frequency for the measured particular direction of measurement. In order to obtain a corresponding three-dimensional characteristics of the sample, the data for sensor scanning system coordinates and oscillation amplitude magnitudes needed for keeping constant the value of the physical quantity featuring interaction of oscillating probe element apex with the sample: oscillation phase, amplitude magnitude, resonance frequency or any other physical quantity are acquired and processed.

For those, skilled in the art it will become clear that in the three-dimensional measurement method according to the invention, as well as in two- and one-dimensional measurement methods with said micromechanical sensor, the axes of the sensor and the axes of the scanning system are set co-axial.

In a third aspect, the present invention relates to a method for producing SPM sensor according to the first aspect of the invention, where on (100) oriented p-type doped single-crystal silicon wafer with a specific resistivity from 0.01 to 20 Ω.cm, or n-type doped with a specific resistivity ranging from 0.003 to 20 Q.cm at least one upper sacrificial layer is formed and afterwards a front surface micromachining by patterned etching is performed. Optionally, the process of surface patterning can be preceded by forming of at least one concave functionalization element with a depth lower than the thickness of the probe portion, and in this case surface micromachining is performed.
aligned to that at least one concave shaped functionalization element. During this front surface micromachining by etching, the sensor body, the microcantilever protruding out of it, the probe member and optionally on-axis placed, concavely shaped functionalization element simultaneously are formed in the same plane, with a depth of the etching equal to the thickness of the probe portion, so that a body and a flat flexible structure with at least one concavely shaped functionalization element are obtained. Afterwards they are subjected to surface micromachining from the back side of the silicon wafer and to a subsequent bulk micromachining and releasing of the entire sensor structure, while simultaneously a planar carrier structure is formed. In a following sensor manufacturing step in the so created at least one concavely-shaped functionalization element an elongated probe element is assembled, via positioning or self-positioning it at the bottom of the concavely shaped functionalization element and is fixed to the bottom and finally the completed sensor is released from the carrying planar structure.

In a preferred embodiment of the method for manufacturing SPM sensors according to the invention, the formed on the front-surface of the wafer at least one concavely shaped functionalization element with a depth less than the thickness of the probe portion is obtained prior to the front surface micromachining process by photolithographic patterning and at least one subsequent etching process.

In another particularly preferred embodiment of the method for manufacturing SPM sensors, a single-crystal n-type doped silicon wafer with a resistivity from 1 to 20 $\Omega \cdot cm$ is used, which in addition contains previously produced doped and galvanically connected highly conductive and piezoresistive areas. In this case during the process of photolithography for front surface micromachining the microcantilever and probe members are aligned to the doped areas. Afterwards, in the subsequent process of front surface micromachining by etching out of the piezoresistive areas piezoresistors are formed. Further on the manufacturing method continues with the processes of metallization, where in contact with the highly conductive areas on the probe member, on the microcantilever and on the sensor body, metal pathways with contact pads are
patterned to connect the piezoresistors with a system for the measurement of the electrical signal.

These and other specific features of the invention, as well as its advantages are described in detail later on in the specification and the examples which illustrate it without restricting it.

**Brief Description of the Drawings**

Fig. 1 shows an axonometric view of a classical microcantilever sensor as known from the prior art in the field of SPM.

Fig. 2 depicts an axonometric view of a microcantilever subject to a force with arbitrary direction as known from the prior art.

Fig. 3 shows a view of a microcantilever sensor with a single measurement direction along the $X$ axis with a functionalization element in the form of V-shaped trench.

Fig. 4 shows a view of a microcantilever sensor with a single measurement direction along the $X$ axis and a probe element making an angle of $120^\circ$ with the axis of the microcantilever.

Fig. 5 is a view of a microcantilever sensor with a single measurement direction along the $Z$ axis with a functionalization element in the form of a right triangular prismatic opening.

Fig. 6 shows a view of a microcantilever sensor with a single measurement direction along the $X$ axis and two probe elements making different angles with the axis of the probe member.

Fig. 7 shows embodiments of sensors from the invention with two measurement directions, and includes Fig. 7A, which depicts a view from above of a sensor with two probe elements along the $X$ and $Z$ axes, Fig. 7B which is a from above of a sensor with two probe elements along the $X$ and $Y$ axes, Fig. 7C which is a from above of a sensor with three probe elements along the $X$ and $Y$ axes, said sensor can perform measurements
along the $Y$ axis in forward and reverse direction, and Fig. 7D which is a view from above of a sensor with three probe elements with different orientation in the $XY$ plane.

Fig. 8 shows an embodiment of sensors of the invention with three measurement directions, where Fig. 8A shows a view from above of a sensor with three probe elements along the $X$, $Y$ and $Z$ axes, and Fig. 8B is a view from above of a sensor with four probe elements along the $X$, $Y$ and $Z$, while the measurement along the $Y$ axis can be performed in forward and reverse direction.

Fig. 9, where Fig. 9A shows an axonometric view of a sensor with three measurement directions with four probe elements in three axes: $X$, $Y$ and $Z$, while the measurement along the $Y$ and $Z$ can be performed in the forward and reverse direction, and Fig. 9B shows the frequency characteristic of the normalized amplitude magnitude of the mechanical oscillations of the sensor from Fig. 9A in three directions.

Fig. 10, where Fig. 10A shows an axonometric view of a sensor with three measurement directions with one probe element and piezoresistive oscillation detection. Fig. 10B presents a principle electrical scheme of the piezoresistors in this sensor, Fig. IOC shows the frequency characteristic of the resistance of the serially connected piezoresistors, Fig. 10D presents examples for probe elements.

Fig. 11 shows the structure of a micromechanical SPM sensor with a functionalization element in the form of an opening formed as a right triangular prism after the front surface micromachining is completed.

Fig. 12, where Fig. 12A, Fig. 12B, Fig. 12C-I and Fig. 12C-II show different embodiments to obtain functionalization elements in the form of trenches.

Fig. 13, where Fig. 13A shows structures of a micromechanical SPM sensor after the front surface micromachining, as aligned to functionalization elements and Fig. 13B shows such structures for the embodiment of a sensor with piezoresistive detection.

Fig. 14, where Fig. 14A depicts the axonometric view of a probe member with a probe portion on which the V-shaped and a composed trench functionalization elements are aligned as shown in enlarged view in Fig. 14B and Fig. 14C.
Fig. 15, where Fig. 15A shows the axonometric view of a probe member, while Fig. 15B shows the cross section of the probe portion during positioning of the probe element within the functionalization element V-shaped trench.

Fig. 16, where Fig. 16A shows a view from above, while Fig. 16B and Fig. 16C show a cross sectional views of the functionalization element with a probe element after positioning attached correspondingly to the two types of functionalization elements in the form of trenches.

**Detailed Description of the Invention**

**A. High precision and high resolution SPM sensor**

An essential feature and advantage of the sensors manufactured in accordance with this invention is that they possess varying in form and orientation microcantilever elements and probe members, while their probe elements are with exact positioning and protrusion lengths from the probe portion with a high aspect ratio, thus providing a sensor resolution in the range of, and better than 1 nm when used for SPM measurements.

Second essential feature of the sensors according to the invention is that they possess micromechanical cantilever type flexible elements, which can be bended as it is desired in one, two or three directions, thus permitting a single sensor to perform measurements with feedback control in each and every of these directions depending on the preferred embodiment. This unexpected and extraordinary advantage of the sensors of the invention presented is explained through the principal model depicted in Fig. 2 and is further demonstrated in the implemented sensors according to the invention. As shown in Fig. 2, when microcantilever 2 is bending due to the action of the force $F$ with components $F_x$ and $F_z$ along axes $X$ and $Z$, this force causes a displacement $\Delta X$ and $\Delta Z$ of its free end along axes $X$ and $Z$ correspondingly. Among these quantities, in analogy to equation (1) following relations are valid:

$$F_x = k_x X, \quad (4)$$

$$F_z = k_z Z, \quad (*)$$
with $k_x$ denoting the stiffness coefficient along the X axis, while $k_z$ is the stiffness coefficient along the Z axis of the microcantilever, both being determined by the geometry and material properties. For a homogeneous microcantilever with a rectangular cross section, in analogy with equation (2), for $k_x$ and $k_z$ following relations are valid:

$$k_x = E \cdot a_z \cdot a_x^3 / (4t^3), \quad (5)$$

$$k_z = E \cdot a_x \cdot a_z^3 / (4t^3), \quad (5')$$

where $a_x$ and $a_z$ are correspondingly the parameters height and thickness of the microcantilever.

In addition, in analogy with equation (3), the microcantilever has resonance frequencies $f_{ox}$ of bending oscillations along the X axis and $f_{oz}$ of bending along the Z axis, which are defined by the relations:

$$f_{ox} = 0.162 \cdot (E/p)^{1/2} \cdot a_x \cdot l^2, \quad (6)$$

$$f_{oz} = 0.162 \cdot (E/p)^{1/2} \cdot a_z \cdot l^2 \quad (6')$$

Under the effect of a force with equal modular components $F_x$ and $F_z$ the displacement ratio between $\Delta X$ and $\Delta Z$ along the X and Z axes is defined through the equation:

$$\Delta X / \Delta Z = a_z^2 / a_x^2 \quad (7)$$

For the ratio of resonance frequencies along the X and Z axes is valid the relation:

$$f_{ox} / f_{oz} = a_x / a_z \quad (8)$$

Therefore, by appropriate determination of the parameters $a_x$, $a_z$ and / predefined values for the mechanical parameters of the microcantilever can be achieved, like stiffness coefficient and resonance frequency along the X and Z directions.

For microcantilevers with a more complex shape sometimes it is convenient to use relevant normalized parameters, such as normalized lengths like $l_x$ and $l_y$, where the relations (5), (5'), (6), (6'), (7) and (8) remain valid and only an appropriate scaling coefficient is added. When needed, for every specific case the relation of the values of the normalized parameters and the real geometrical dimensions can be derived. For those, skilled in the art it will be clear, that in the present specification when describing the
principles on which the invention is based, both above mentioned kind of parameters are used.

Similarly, when measurement in a direction of the Y axis is desired, through orienting the flexible micromechanical elements along the X axis and presetting of their width and thickness parameter values an oscillation displacement of the probe portion with pre-defined characteristics along the Y and Z axes is realized.

For sensors of the present invention, which have flexible elements oriented along both X and Y axes, placed in common flat surface, by presetting of the relevant geometrical parameters, probe portion displacement with pre-defined characteristics along X, Y and Z directions can be achieved.

In addition, it was surprisingly found, that the flat flexible micromechanical element manufactured according to the presented invention, which has two or three degrees of bending freedom and is capable of performing measurements with feedback control in two or three measurement directions respectively, under periodical actuation with varying frequency within a frequency range which includes a resonance frequency, converts said actuation in bending oscillation in the direction where the resonance occurs. In this manner, having one sensor and one single-axis harmonic actuator samples along two or three measurement directions can be investigated, as each direction of resonance deflection is unambiguously determined by an individual resonance frequency.

Therefore, using said sensors, capable to bend in two or three directions object of the presented invention, measurements with feedback control are performed in an extremely simplified and rationalized manner, as in every point of measurement with the variation only of the actuation frequency and/or the actuation amplitude magnitude, without need to rotate the sensor and/or sample and without need to exchange the sensor, all directions in which the flexible micromechanical element is able to bend are investigated. Furthermore in distinction of the existing SPM sensors, which interact with the investigated sample in a direction orthogonal to the plane of their substrate surface, the sensors manufactured in accordance to the method described in this invention can interact
as in a direction orthogonal to the plane of their substrate surface and in directions parallel to the surface of their substrate as well, i.e. as needed, they are sensitive along one, two or three axes.

Another advantage of the flat sensors obtained in accordance to the method of the present invention is the opportunity of placing heterogeneous probe elements with appropriate shape and features in direction at will in the plane of the substrate, as well as perpendicular to this plane. In addition, the need for etching a dominant part of the surface in order to shape a tip drops out, hereby creating the unexpected opportunity of producing sensors with highly reproducible parameters through a simplified process sequence using materials as well as equipment which are common for the field. Further, reproducibility of sensor parameters is enhanced due to the precise control of the positioning and orientation processes and the desired optimum protrusion length of the probe elements out of the probe portion.

A substantial advantage of said sensors according to the present invention is the opportunity of implementing various probe elements placed in the same functionalization elements while the type of the probe elements is of no importance; decisive are their geometrical parameters only.

B. Method for three-dimensional measurement

In the state of the art methods for SPM, during measurement the sensor moves relatively to the sample by tracking a grid of points, dependent on the scanning system algorithm employed. The grid of points is defined by incremental steps $\delta_h$ equal or differing for each scan direction. Tracking is usually accomplished in the method of the present invention as well. According to this invention, during three-dimensional measurements, the employed sensor with a flexible micromechanical element, which possesses the features as described in the present invention, is driven into excited oscillation of bending without torsion. Characteristic of this process is that the magnitude of oscillation amplitude of the probe portion of a micromechanical flexible element
depends on the intensity of the periodic actuation on the sensor. On the other hand, other things being equal, the magnitude of excited oscillation amplitude of a flexible element, oscillating with varying frequency in the range of its resonance frequency is significantly, i.e. up to hundred times, bigger than that outside of said frequency range. In other words, for a fixed intensity of a periodic actuation, while varying only the frequency of the excited oscillation of the flexible micromechanical element which is bendable in all three directions, only at frequencies close to (or within the range of) any resonance frequency, it will oscillate with an increased magnitude of amplitude in the corresponding direction. Therefore, at a sufficiently close proximity of the probe element apex from the sample, an interaction between them occurs only within a particular of said resonance frequency ranges. As a result of the interaction, a perturbation of the oscillation parameters and/or a tunneling current can be detected. In this case, an unambiguous relation exists between the frequency at which the interaction is detected and the direction in which the interaction occurs - this direction is known in advance, as it is the one in which at that particular frequency the flexible micromechanical element is in resonance.

The method for three-dimensional measurements with feedback control according to the present invention, employs an excited bending oscillation of the flexible element in two substantially distinct in magnitude amplitudes: a big magnitude "test amplitude", used for the initial detection of interaction between the probe element and the scanned sample surface and a "working amplitude" being ca. 10 to 200 times smaller than the "test amplitude", with 100 times smaller being the preferred magnitude, which is used during surface scanning with a high resolution. Both said amplitudes are obtained through a setting of the intensity and/or frequency of the periodic actuation.

The relation between the amplitude of excited oscillation and its frequency for a preset intensity of the actuation is described by a continuous function. This allows determining the difference $M_i$ between the amplitudes of excited oscillations with a frequency equal to the resonant and with frequencies outside the range of resonance in the $i$-th measurement direction and normalized relatively to the actuation intensity. The
value of this difference $M_i$ for each measurement direction is characteristic for the sensor and is used to determine the translation incremental step modulus $A_i$ of the scanning system in the direction of the initial approach to the surface, while the following relation is obeyed:

$$A_i = \epsilon \cdot M_i$$ \hspace{1cm} (9)

where $\epsilon$ is a coefficient characteristic for the relation between the translation step $A_i$ of the scanning system and the difference $M_i$ between the magnitudes of amplitudes in any direction $i$ while incidental contact is avoided.

At high resolution surface scanning with incremental steps of $\epsilon_i$ in each of the three possible directions, the intensity and/or frequency close to the resonance one of the exited oscillation are selected in such a way as to perform an oscillation with controlled amplitude.

When a measurement with feedback control with a sensor capable to bend in all three directions is performed according to the three-dimensional measurement method as described in this invention, two measurement regimes are employed successively:

1. A regime for approaching a point of measurement, where during the translation towards the sample the sensor is brought into an exited oscillation in frequency ranges including the resonance frequencies for each of the scan directions $X$, $Y$ and/or $Z$. Initially the magnitude of amplitude of this oscillation is altered until a typical test magnitude for the measurement system used is reached and under the condition that no contact with the sample would happen. Simultaneously with the oscillation the sensor starts to translate relatively to the sample with incremental steps $A_i$ until an interaction is detected. After that by means of the frequency range of the resonance oscillation the direction of the detected interaction is defined. Then, decreasing the magnitude of amplitude down to working one and following the direction of detected interaction $j$, translation continues until an interaction with the sample is detected again. The reached point is a point of measurement, and

2. A measurement regime, in which probe element apex is located at the point of measurement and every point within an area with size $(2d_j)(2S_{j+1})$ around said point of
measurement in parallel to sample surface is investigated while the sensor and the sample
are in a fixed state without movement or rotations. For the purpose, after the reaching of
the measurement point, in the measurement direction \(j\) oscillations with working
amplitude and a frequency range containing the resonance frequency for this direction are
performed again, and the physical quantity value featuring the interaction of the probe
element with the sample surface is determined. After that the area around the point of
measurement is checked in \(j\) directions by bringing the sensor into an exited oscillation
with altering from working to test magnitude of amplitude in a frequency ranges
including the resonance frequencies for each of the said remaining directions. Later on in
directions with no detected interaction following a preferred tracking algorithm, scanning
continues with incremental steps \(\delta_j\), like the described for the first direction, while
simultaneously data for the perturbation of phase, magnitude of amplitude, resonance
frequency or other measured physical quantity as resulting from the interaction with the
sample are acquired and processed, in order to obtain the corresponding three-
dimensional characteristic of the sample.

A substantial and unexpected advantage of the measurement method described
above is that in any point of measurement oscillations with different magnitudes of
amplitude can be applied - from working to test and without rotating or exchanging the
sensor, through variation of the intensity of the periodic actuation and/or the range of the
frequencies in a single point, because of the feedback control all directions via oscillation
of the flexible micromechanical part with the probe element/elements can be investigated
with a resolution better than that determined by incremental steps \(\delta_i\). Furthermore the
necessary data for the investigated object, as well as for performing an optimized
algorithm of sample scanning are obtained. In this manner, according to the method for
the three-dimensional measurement of the present invention, a sample can be investigated
entirely in each point during a single, simplified and convenient measurement process
with one and the same sensor. Simultaneously optimized flexible measurement
algorithms are employed, which are not restricted by the form of the samples and the
measurement is accomplished without the necessity of a priori information to be available as is required in nowadays methods, which depends on parameters such as pattern pitch, profile steepness, form of undercutting etc. to be known approximately.

Thus, through the three-dimensional measurement method with said sensor of the present invention, which employs in each point of the scan grid an actuation with varying both frequency and magnitude of amplitudes, by bending oscillation in different directions samples with a surface in any measurement direction \( j \) and with a profile steepness restricted only by the ratio of the mentioned amplitude difference \( M_i \) and scanning step in transverse direction \( \delta_{+1} \) are scanned and characterized.

C. Method for manufacturing sensors for scanning probe microscopy

The main technological and systematic advantage of said method for the manufacturing of sensors according to the invention is that as the body, microcantilever and probe member are flat and obtained in the substrate surface, said method allows conventional photolithographic patterning to be employed, thus a variety of, including several probe elements on a single microcantilever sensor can be produced in accordance to the intended application of the sensor and the entire measurement system used. Furthermore, important parameters of the micromechanical elements obtained through this method, namely \( a_x, l_x, a_y \) and \( l_y \), are determined by a single photolithographic patterning and are not dependent on the misalignment between different photolithographic layers. As far as the methods of photolithography used in practice provide sufficient uniformity on wafer and wafer-to-wafer reproducibility, this creates opportunity sensors used in high resolution SPM to be manufactured with reproducible characteristics.

Another important advantage of the method described above for the manufacturing of microcantilever or other flexible elements of micromechanical sensors is that it creates opportunity elements with different proportions between their geometrical characteristics \( a_x, l_x, a_y, l_y \) and \( a_z \) to be manufactured simultaneously, thus to obtain sensors possessing a
measurement direction in any of the axes X, Y and Z with a correspondingly different
oscillation frequency ranges.

Another advantage of said method presented in the invention is, that as in the
manufacturing process of the sensors no convex structures are present, probe elements
are assembled in a reproducible manner into functionalization elements of the sensor
through non-critical positioning or self-positioning and a subsequent fixation processes.
At the same time, said method allows probe elements with high aspect ratios to be
employed, while their protrusion length outside the probe end \( l_p \) varies in a wide range
without the need of a different treatment of the sensor structure.

Example 1 - Sensor with a Single Measurement Direction

Fig. 3 depicts an example of a preferred embodiment for the implementation of a
SPM sensor according to the present invention with a single measurement direction
consisting of a body \( I \), a protruding out of it microcantilever \( 2 \) and a probe member \( 3 \).
The probe member \( 3 \), which protrudes at length \( l \), out of body \( I \), with a probe element \( 4 \)
allows interaction through a surface, which is perpendicular to the plane of the wafer.

The microcantilever \( 2 \) according to the invention has a connecting microcantilever
part \( 5 \), which joints it to the body \( I \) and is of height \( a_x \) and can be smaller, such as it is in
the present example or it can be equal to the height of the microcantilever \( 2 \). The free end
of the microcantilever \( 2 \) is joined with the probe member \( 3 \) which is protruding out of it,
while the axes of these two elements making angle \( \beta \) of 90°, intersect in point \( C \), which
determines the characteristic length \( l_x \) of the microcantilever as the distance of the
intersection point \( C \) to body \( I \). When above mentioned two elements are coaxial, the
position of point \( C \) is defined by the position of probe element \( 4 \).

The probe member \( 3 \) has a probe portion \( 6 \), in which a functionalization element \( 7 \)
in the form of a V-shaped trench having depth of \( d_y \) is arranged, crossing the surrounding
sidewall \( 8 \).

The body \( I \), the microcantilever \( 2 \) and probe member \( 3 \) with the probe portion \( 6 \)
are formed simultaneously out of a common single-crystal semiconductor silicon
substrate in one plane, representing their common flat surface having no convex members. For this flat microcantilever 2 it is featured that where its geometrical characteristics are obeyed of the relation:

\[ a_z \geq 2 \cdot a_x \]  

(10)

wherein \( a_x \) is the height of the microcantilever connecting part 5 and \( a_z \) is the thickness of the microcantilever 2, then this microcantilever, according to the relations (7) and (8) has a bigger deflection \( AX \) and accordingly, a lower resonance oscillation frequency along the \( X \) axis in comparison to along the \( Z \) axis. This gives opportunity by presetting of suitable values of the parameters \( a_x \), \( a_z \) and \( l_x \), a flexible micromechanical element in a frequency range of measurement to have only one resonance frequency and thus one direction of measurement along the \( X \) axis, in which direction of measurement probe element 4 is provided.

In accordance with the present invention, the probe elements 4, which are used in the sensors, are individual, ready elongated elements with a small radius or cross section size and with an aspect ratio from ca. 1:10 to 1:5000. These probe elements are heterogeneous - they are made of a material different from the material of the substrate, which could be carbon, i.e. a single- or multiwall cylindrical CNT, but other elongated heterogeneous carbon, boron, boron nitride, or metal elements, i.e. nanowire, nanofibre, nanorod, or zinc-oxide micro- or nano-crystal etc., as was mentioned already above, can be implemented as well. Any kind of elongated heterogeneous elements or their bundles are applicable, including elements of complex configuration, i.e. a nanotube ending with a sphere, a pyramid, multi-pyramidal shape etc. and the heterogeneous probe elements can be pretreated appropriately as well, in order to acquire certain physical, chemical other desirable properties. In the present example the probe element is an electrically conductive single-wall CNT, with aspect ratio of approximately 1:1000. In other preferred embodiments of the invention the application of bundles of CNTs are favored.

The individual elongated heterogeneous probe element 4, which is attached in the V-shape trench functionalization element 7, crosses one-sidedly the surrounding sidewall
8 of the extended part of the probe portion 6. The V-shaped trench is obtained as a result of aligned in the <110> direction wet etching mask window during process of trench formation into the single-crystal silicon substrate. The orientation of the probe element 4 coincides with the orientation of the V-shape trench functionalization element 7 and the positioning accuracy of said probe element is determined from the precision of manufacturing the functionalization element.

This variant of implementation of the sensor is especially applicable for the investigation of samples, for which it is of importance the probe element to be orthogonal to the investigated sample surface.

Another embodiment of a sensor with a single measurement direction is shown in Fig. 4. It comprises of a microcantilever 2 with a characteristic length $l_x$ and dimension $a_x$ of the connecting microcantilever part and a probe member 3 with a probe portion 6. In this embodiment the probe member 3 and the functionalization element - a composed trench 9 with depth $d_t$ are oriented such that the axis of the probe member 3 forms an angle $\beta$ with the axis of the microcantilever 2, which can be varied in the range of 30° to 180°; while in said embodiment the preferred angle $\beta$ equals 120°. This implementation variant of the sensor is especially applicable for the investigation of samples, for which it is of importance that the sample area interacting with the probe is under direct visual observation.

Fig. 5 shows another probe embodiment according to the invention, with a probe element 4, assembled perpendicularly to the common flat surface of the sensor in a functionalization element opening 10 and protruding out of the said surface at length of $l_{pz}$. Similarly, the sensor comprises of body 1, a protruding out of it microcantilever 2 joined with body 1 through the microcantilever connecting part 5 and a protruding out of the microcantilever probe member 3 with a probe portion 6, which are situated in one plane, being their common flat surface not possessing any convex portions. For the flat microcantilever 2 it is featured that where its geometrical dimensions obey the relation:

$$a_x \geq 2 \cdot a_z$$  \hspace{1cm} (11)
wherein $a_x$ is the width of the microcantilever connecting part 5, while $a_z$ is the thickness of the microcantilever 2, then this microcantilever according to the relations (7) and (8) has a bigger displacement of the probe portion $AZ$ and correspondingly, a lower resonance frequency along the $Z$ axis in comparison to those along the $X$ axis. This makes it possible, in a frequency range of measurement through presetting of suitable values for the parameters $a_x$, $a_z$ and $l_z$, of the flexible micromechanical element, to have only one resonance frequency and correspondingly, one measurement direction along the $Z$ axis, in which the probe element is oriented.

The orientation of probe element 4, as shown in the detailed axonometric view in Fig. 5 is determined by the slope of the sidewalls of the right triangular prism opening shaped functionalization element 10, determined by the etching process. As it is known to those, skilled in the art such etching methods exist, as for example the so-called Bosch process, where a reproducible vertical slope with an accuracy better than $\pm 2^\circ$ is obtained. Besides, any other form of opening, which allows positioning or self-positioning of probe elements, can be used as well.

Fig. 6 illustrates a sensor embodiment, which despite being provided with two composed trench functionalization elements 9 and 9′ and two probe elements 4 and 4′, has an entirely different implementation purpose, based on the fact that the probe elements have a common imaginary intersection point $A'$ of their axes, which is located outside the boundaries of the probe portion 6 in the measurement direction of the sensor. When this intersection point $A'$ is the point of measurement as well, such a sensor is suitable for simultaneous measurement of two sample characteristics, or for measuring the influence of the presence of one of the probes on the signal of the other, as well as for measuring the influence of the presence of a sample on the interaction between the probes. For this purpose, the two probe elements 4 and 4′ being arranged to form angles $\Theta$ and $\theta'$ equal to correspondingly 51° and 37° towards the axis of the probe member, are of distinct type and properties, when the sensor is dedicated for the measurement of two different sample characteristics, although they can be equal when the method for the
investigation of the sample is based on its influence upon the interaction among the two
probes. Like in the previous implementation, the protrusion lengths of probe elements 4
and 4' are preset in advance. In this example the distinction of the interaction among both
probe elements is not necessary as the measurement is one-dimensional.

**Example 2 - Two-Dimensional Measurement Sensor**

Fig. 7A depicts an embodiment of a sensor from the invention which has a
microcantilever with a characteristic length \( l_x \) in the \( Y \) direction and height \( a_x \) of the
microcantilever connecting part 5, a probe member 3 with a probe portion 6 and is with
two functionalization elements: a V-shape trench functionalization element 7 and an
functionalization element opening 10.

For this sensor embodiment it is featured, that the relations (10) and (11) are not
fulfilled simultaneously and the values of two parameters \( a_x \) and \( a_z \) are approximately
equal, but nevertheless different, i.e.:

\[
a_x \approx a_z, \text{ although } a_x \neq a_z \tag{12}
\]

wherein \( a_x \) and \( a_z \) are the height and thickness of the microcantilever as shown in
Fig. 7A and the accompanying enlarged detail.

In this embodiment, by means of the appropriately determined characteristic
length \( l_x \) and height \( a_x \) the desired oscillation properties of the sensor are obtained, with a
microcantilever 2 having approximately the equal deflection along the \( X \) and \( Z \)
directions. Including, the parameters are determined in a way to fulfill the relation:

\[
\gamma \cdot Q \cdot (f_{ox} - f_{ox}) \geq f_{ox} \tag{13}
\]

wherein \( \gamma \) is a coefficient in the range from 0.1 to 10, preferably 0.2 to 1.0, which
features the method for recognition of the measurement direction based upon the
frequency distribution of a certain parameter (i.e. the amplitude of the excited oscillation)
when the micromechanical element is bended, which is known in advance for any
specific measurement, \( f_{ox} \) is the resonance frequency of the microcantilever 2 bending
along the \( X \) direction, \( f_{oz} \) is the resonance frequency of the microcantilever 2 bending in
the \( Z \) direction and \( Q \) is the average value of the quality factors of the microcantilever for
bending in directions $X$ and $Z$. Thus, the microcantilever 2, with a probe member 3, for which the relations (12) and (13) are fulfilled simultaneously has the resonance frequencies $f_{\alpha x}$ and $f_{\alpha z}$ of bending along the $X$ and $Z$ axes, which are of nearly equal value, but are unequivocally discernable. Accordingly, a sensor with such a construction of the microcantilever 2 and its probe member 3 with two probe elements 4 and 4' as shown in Fig. 7A, has two measurement directions along said axes thus allowing the conduction of two-dimensional measurements in any point of the scan grid.

Additionally, every deflection of the microcantilever 2 and displacement of its probe member 3 in the $XZ$ plane can be expressed as an equivalent sum of two independent components along the two mutually perpendicular measurement directions $X$ and $Z$. Any oscillation at the frequency $f$ of said microcantilever can be interpreted as sum of two independent oscillations with the same frequency along the two axes with the amplitude magnitude ratio of the two components being constant for every frequency $f$, which is outside the ranges of the resonance frequencies $f_{\alpha x}$ and $f_{\alpha z}$. Accordingly, with two measurement directions in the preset measurement frequency range, the magnitude of the normalized oscillation amplitude in the direction for which a resonance is present is bigger than that in the directions with no resonance. Thus, when placed in a measurement direction, the probe element interacts with the investigated object in a different way compared to the interaction in another direction, accomplished by another probe element, arranged on the same microcantilever. As a result, the parameters of the oscillation are perturbed and/or a tunneling current is detected.

In this embodiment the axes of the probe member 3 and of the functionalization element, in which the probe element is oriented towards the $X$ axis, are aligned towards the direction $<110>$ of the substrate, that is why the functionalization element 7 is a V-shape trench with depth $d_{p}$ while the other functionalization element opening 10 is a right triangular prism, as it is shown in Fig.7A. When the alignment of said functionalization element for measurement along the $X$ axis is different, the functionalization element will be a composed trench.
Another preferred embodiment for the implementation of the invention is shown in Fig. 7B. In this case the sensor has a microcantilever 2, a probe member 3 with perpendicular axes with two V-shape trench functionalization elements 7 and 7'. These elements are placed on the probe portion 6 of the probe member 3, which in this variant is shaped with an additional extended region placed in the same plane. The V-shape trench functionalization elements 7 and 7' are oriented so that the axes of both probe elements 4 and 4' having the imaginary intersection point A which falls in the central part of the probe member 3, are arranged under the angles Θ and Θ' towards the probe member 3, which can be from 0° to 180° respectively.

Considered alone, the probe member 3 with the probe connecting part 11 with rectangular narrowing of width \( a_y \), a probe portion 6 with an additional extended region of length \( l_y \), defined as the distance from point A to the beginning of the microcantilever 2, for which is fulfilled the relation:

\[
a_z \geq 2 \cdot a_y \tag{10'}
\]

has a preferred bending along the Y axis and correspondingly a lower resonance oscillation frequency along this axis in comparison to these along the Z axis, which permits measurements along the two axes to be recognized in amplitude and frequency. With a thickness \( a_z \) the oscillation characteristics for this sensor embodiment according to the invention, including bending resonance frequency \( f_{o\alpha} \) of its probe member 3 along the Y axis, are determined by the width \( a_y \) and the characteristic length \( l_y \).

Thus the microcantilever 2 with the probe member 3, shown in Fig. 7B, for which both the relations (10) and (10') are met and with appropriately preset parameters \( a_x, a_y, l_x \) and \( l_y \), will have close but discernible resonance frequencies along the X and Y axes. Additionally, every deflection in the XY plane can be expressed as equivalent sum of components along the two mutually perpendicular measurement directions, X and Y correspondingly. Therefore, within a particular measurement frequency range and with suitably determined parameters \( a_x, a_y, l_x \) and \( l_y \) a flexible micromechanical element with different, including close values for the resonance frequencies \( f_{o\alpha} \) and \( f_{o\alpha} \) of the bending
oscillations along the X and Y directions is obtained. Any oscillation with frequency can be considered as a sum of two independent oscillations with the same frequency in both directions, while the amplitude magnitude ratio of both components is constant for every frequency, which is outside the ranges of the resonance frequencies $f_{ax}$ and $f_{ay}$ of the mechanical oscillations. Accordingly, with two measurement directions in the preset measurement frequency range, the magnitude of the normalized oscillation amplitude in the direction for which a resonance is present is bigger than that in the directions with no resonance. Thus, in the direction which is the measurement direction, the probe element interacts with the investigated object in a different way compared to the interaction in another direction, accomplished by another probe element, placed on the same microcantilever.

When the axes of the functionalization elements are aligned towards the direction <110> of the silicon substrate, the shape of the cross section of these two functionalization elements are V-shaped trenches 7 and 7'. Because the probe elements 4 and 4' are heterogeneous and no restrictions on their type exist, they can be identical or different in type and dimension. In this particular example, as shown in Fig. 7B, the lengths of the probe elements 4 and 4', which are single wall CNTs, are such that they protrude out of the probe portion to preset distances $l_{px}$ and $l_{py}$ from the imaginary intersection point A of their axes, oriented so that the point A to fall in the central region of the probe member 3.

Thus, it is feasible to employ said sensor to scan and characterize samples with variable surface in any of the X and Y directions with a profile steepness limited by the ratio of the amplitude difference in the corresponding direction and the incremental scan step in the transversal direction, as described above.

In Fig. 7C a third embodiment of the invention in two measurement directions, is shown. In this case the sensor has a microcantilever 2 and a probe member 3 with perpendicular axes and three functionalization elements arranged, in parallel to the substrate plane, in the probe portion 6 which accordingly has two additional extended
areas. The axes of the three probe elements 4, 4' and 4'' having the imaginary
intersection point A, are located under angles θ, θ' and θ'' towards the axis of the probe
member 3 and are correspondingly 90° or 180°. As the axes of functionalization elements
are aligned in the <110> direction of silicon substrate, they are V-shaped trenches,
respectively 7, 7* and 7''. Although this sensor has three probe elements, it can measure
in two directions; hence, two of the probe elements are coaxial, but are in a mutually
opposing direction along the Y axis.

A fourth embodiment for the implementation of the invention with two
measurement directions is shown in Fig. 7D. In this case the sensor has a microcantilever

and a probe member 3 with perpendicular axes and three functionalization elements
located in parallel to the substrate plane, on the probe portion 6, which correspondingly
has two additional extended areas. The functionalization elements are oriented so that the
axes of the three probe elements 4, 4' and 4'' having the imaginary intersection point A,
are located under angles θ, θ' and θ'' towards the axis of the probe member 3 and can be
from 0° to 180° accordingly. When at least one of the functionalization element axis is
aligned in a direction different from <110> of the silicon substrate, then the
functionalization elements are composed trenches 9, 9' and 9'' which can be arranged in
a metal layer as well. Although this sensor has three probe elements and non two of them
are coaxial as in this embodiment, it can measure in two directions, X and Y respectively.

Example 3 - Sensor with Three Measurement Directions

Fig. 8A shows an embodiment of the sensor in accordance with the invention with
three probe elements. The probe elements 4 and 4' are arranged in a plane parallel to the
plane of the substrate, similar to the sensor shown in Fig. 7B, while the probe element
4'' is perpendicular to said plane. The microcantilever 2 is with a characteristic length \( l_x \)
in the Y direction and height \( a_x \) of the microcantilever connecting part 5 and has a probe
member 3 with a connecting part 11 with a rectangular narrowing of width \( a_y \) and the
characteristic length \( l_y \). On the probe portion 6 two additional extended areas are shaped
and three functionalization elements are created, two of which are V-shaped trenches 7
and 7', and one is opening 10, all oriented in such a way, that the axes of the probe elements 4, 4' and 4'' form with the axis of the probe member 3 angles 0, 0' and 0'', which are equal to 180° or 90° accordingly. When is necessary or preferred, functionalization elements to be V-shaped trenches, their axes are aligned correspondingly in the direction <110> of the silicon single-crystal substrate. Besides, the three probe elements 4, 4' and 4'' protrude outside of the trenches, respectively in any of the directions X, Y and Z at the distance l_{pz}, l_{py} and l_{pz}. As in Example 2, the heterogeneous probe elements can be the same, including in respect to their electrical conductivity or they can differ in type or dimension as the length of their protrusion are preset in relation to the imaginary intersection point A of the axes of the probe elements 4, 4' and 4''. As general preferred option, different types of heterogeneous probe elements are appropriate to be used when investigating morphology and orientation of magnetic domains or thin multilayer structures with different properties of the individual (sub)layers, such as electric conductivity, magnetic permeability, leading to different properties of the structure in the respective directions.

Fig. 8B and Fig. 9A show an embodiment of a sensor with four probe elements located in three mutually perpendicular axes. In this case also, since the axes of the functionalization elements are aligned in the <110> direction of the single-crystal silicon substrate the functionalization elements are V-shaped trenches, 7, 7' and 7'' respectively.

Because of the opportunity for precise control of the position of each of the functionalization elements and the probe elements, provided for the present invention, they can be obtained so that the axes of the V-shape trench functionalization elements 7, 7' and 7'' to have a common imaginary intersection point A, in which an functionalization element opening 10, representing right triangular prism is formed, positioned so that the axis of probe element 4'', placed into it to pass through said intersection point A. Besides, the probe elements 4, 4', 4'' and 4''' protrude beyond the functionalization elements, respectively, along each of the axes X, Y, Z, as Y and Z axes can be measured in the forward and reverse direction. The probe element ends, as in the
previous cases, are at the distances of lengths $l_{px}$, $l_{py}$, $l_{pz}$ and $l_{p-z}$ defined regarding the imaginary intersection points of the four functionalization elements' axes. Similarly, the heterogeneous probe elements 4, 4' 4" and 4" can be selected to be identical, including their electrical conductivity, or be of different length and type.

Fig. 9A shows an axonometric view of a sensor for scanning in the $X$, $Y$ and $Z$ directions, whose heterogeneous probe elements can have equal or different protrusion lengths from the common imaginary intersection point of their axes to the end of the protruding probe elements. Since the distance from the intersection point to the end of the probe element and the distance of interaction between the probe element apex and the sample are known, thus it is possible to calculate the geometric shape of the investigated sample in the respective direction with high accuracy. Likewise, the detected signal is used for adjusting both translation and actuation amplitude magnitude of the sensor in the measurement direction as well, to maintain a constant value of the physical quantity characterizing the interaction with the sample, which in this example it is the phase of the exited oscillation, but can be the amplitude or the resonance frequency as well.

Fig. 9B shows the frequency dependence of the normalized amplitudes' differences $M$. Similarly to previous examples, frequency at which a perturbation in the mechanical oscillation or tunneling current is detected is used for recognition of the direction in which interaction between the probe element and the sample is achieved.

This sensor is suitable for probe investigation of three-dimensional samples with a complex form.

Surprisingly for the sensors of this invention it was found that by means of a single flexible micromechanical element the surface morphology or composition of one-dimensional, two-dimensional or three-dimensional objects can be determined, wherein the dimensions of measurement coincides with the number of the measurement directions. Whereas through variation of the microcantilever geometrical parameters in the different directions the oscillation characteristics of the sensors in any of these
directions can be pre-determined; their eigenfrequencies in these directions can be set so that they to be close, but unambiguously discernible.

In Fig. 10A an axonometric view of a sensor for SPM with three measurement directions is shown, similarly to the one shown in Fig. 9A, which has only one heterogeneous probe element, selected alternatively among heterogeneous probe elements 4, 12 or 12’ shown in Fig. 10D. To detect the interaction of this probe element in any of the three measurement directions, piezoresistive sensor elements are used, which include five highly doped areas 13 realizing galvanic contact with the respective serially connected areas with piezoresistive properties 14, 14’ and 14”. These piezoresistive sensor elements alter the value of their resistance when the probe portion is displacing in the corresponding direction X, Y and Z. In the embodiment as shown in Fig. 10A, the three piezoresistors 14, 14’ and 14” are connected serially through four contact holes 75 and by means of three metal paths 16 with contact pads 77 and 17’. In this connection variant only the resulting sum of electrical resistance values of the three piezoresistors is measured using an external measurement instrument, but depending on the implementation of the sensor, it is clear that other connection approaches are possible as well.

Fig. 10B illustrates the electrical connection diagram of the three piezoresistors as shown in Fig. 10A, with the frequency dependence of the root mean square (RMS) value of the of multi-element resistor $R_{177}$, measured between the pins 77 and 77’ and shown in Figure 10C. As seen, this resistance has three peaks corresponding to the resonance bending frequencies of the micromechanical sensor. At equal oscillation amplitudes in differing directions, these maxima may be equal or different depending on the parameters of piezoresistors, their location on the flexible elements and the method of manufacturing. Regardless of the proportions of individual resistors change, through measuring of said resistor $R_{177}$, the micromechanical elements oscillation perturbations described above, resulting from the interaction of the probe element and the sample in any measurement direction can be detected.
The variations of the heterogeneous probe elements as shown in Fig.10D illustrate some preferred examples, such as an heterogeneous probe element 4, representing a CNT with a hemispherical end, 12 is heterogeneous probe element consisting of joined cylindrical and spherical parts, 12' is heterogeneous probe element composed of a cylindrical part connected to a micro/nanocrystal with specific properties, and pyramidal or multi pyramidal shape.

The sensor resolution with probe element 12 is determined by the diameter of the sphere and such a sensor is suitable for measuring the dimensions of objects with an unknown three-dimensional shape. The resolution of a sensor with probe element 12' is determined by the curvature radius of the pyramid tips of the micro/nano crystal.

Similarly to previous examples, at the frequency at which by measuring the total resistance a perturbation in the oscillation characteristics or the occurrence of tunneling current is detected, recognition of the direction of interaction between the probe element and the sample is achieved. Since the protrusion length of the probe element beyond the functionalization element is known, this enables when measuring with this sensor to determine the geometric shape of the investigated sample in respective directions, with a resolution determined by the diameter of the probe element.

In addition, as another preferred embodiment of the invention, each of the sensors described in Examples 1, 2 and 3 and illustrated in Figures 3, 4, 5, 6, 7A, 7B, 7C, 7D, 8A, 8B and 9A can be manufactured with piezoresistors further integrated onto the surface or in the sidewalls of their flexible micromechanical elements, through the appropriate adaptation, known to those skilled in the art, and dependent on the particular design of the sensor in the manner disclosed above.

Example 4 - Method for Three-Dimensional Measurement with Feedback Control with a Sensor for Scanning Probe Microscopy

At the beginning of the process of a three-dimensional measurement with feedback control, a SPM system provided with a sensor for three measurement directions, which has one microcantilever and one probe member having a common flat surface,
with individual oscillation characteristics of bending without torsion in each of the directions X, Y and Z, which are discernible from one another during detection, is placed in starting position relative to the sample. The probe member of said sensor has to be equipped with a sufficient number of heterogeneous probe elements to ensure measurement in any of the directions X, Y and Z. This can either be a sensor with a single heterogeneous probe element, which is sensitive in the three areas of measuring X, Y and Z, as for example is the sensor of the present invention shown in Fig. 10A, or another type of sensor, such as variations of the sensors from the invention in which the probe member can be provided with three or four heterogeneous probe elements - like the sensors, shown in Fig. 8A, 8B and 9A.

The system is set in a regime of approaching a point of measurement from the scan grid and firstly being brought into excited oscillation with magnitude of amplitude, which is varied until reaching the typical test magnitude of amplitude for the system and then simultaneously translation is started. When the first interaction between the probe element and the sample is detected, depending on the frequency range in which this interaction is detected, its direction j is determined, where j is X, Y or Z, and the excited oscillation with that amplitude is terminated. Then the translational approach regime continues in the established direction of interaction j with amplitude decreasing to working until a measuring point is reached, where the translation of the sensors and/or sample by the scanning system is stopped. The position thereby reached by the sensor probe element apex is the point of measurement, where sensor and sample remain static, and measuring regime with feedback control along the direction of reaching the point is proceed. For this purpose, the sensor is subjected to periodic actuation with working amplitude in the direction j and the value of physical quantity, featuring the interaction is determined. Then, the sensor is set into oscillation mode in the perpendicular directions j±l as the intensity and/or frequency range of actuation is changed, in a way that the amplitude of oscillation in these directions varies smoothly from working to test, thus checking for an interaction with the sample. If there's no interaction detected in any of
directions \( j \neq l \), while keeping the selected physical quantity values constant by feedback control, the checked sample area is scanned according to the tracking algorithm. Through analysis of the feedback signal controlling the actuator, the properties of the sample surface in the respective direction are investigated. The physical quantities featuring the interaction between the probe element and the sample which can be perturbations in oscillation phase, amplitude magnitude, resonance frequency or other measured physical quantity, such as the tunneling current, are measured and processed to obtain the corresponding three-dimensional characteristics of the sample.

In a preferred embodiment of the method for a three-dimensional measurement with feedback control of the investigated sample, the physical quantity magnitude characterizing the interaction is kept constant by controlling the intensity and frequency of the periodic actuation.

**Example 5 - Method for Manufacturing of a High Resolution SPM Sensor**

The method for manufacturing of SPM sensors according to this invention contains three stages and in it as substrates for obtaining the sensors are used either p-type doped silicon wafers with (100) orientation, with a specific resistivity in the range of 0.01 to 20 \( \Omega \cdot \text{cm} \), or n-type doped wafers with specific resistivity in the range of 0.003 to 20 \( \Omega \cdot \text{cm} \) and for all of them the primary flat is \([1,10]\) orientated.

P-type doped single-crystal silicon wafers are used for manufacturing sensors according to the present invention which are designed for use in SPM systems with optical or capacitive detection of the interaction between probe element and sample or in systems which featuring tunneling current.

When SPM sensors with piezoresistive detection are aimed, n-type doped single-crystal silicon substrate wafers with specific resistivity in the range of 1 to 20 \( \Omega \cdot \text{cm} \) are used, and on them highly doped surface areas are created in advance, which areas can be obtained by various methods known to those skilled in the art. They are in galvanic contact with piezoresistive areas located on the surface and/or in trenches' sidewalls,
manufactured for example by the method disclosed in a International Patent Application No. PCT/BG20 10/000007.

Before processing, each wafer according to the method of this invention, should be pretreated, as it is usual in the field, including chemical treatment as known, to carry on subsequent oxidation processes, etching, etc. as described below in the example of the method implementation.

**Surface micromachining**

According to this invention, at the first stage of the method the substrate is subjected to surface micromachining processes. For this purpose, initially a double-side oxidation of the silicon wafer 18 is carried out, thus forming a silicon dioxide layer 19, being used as a mask in the subsequent processes of surface and bulk micromachining as shown in Fig. 11. Thereafter, the treated silicon wafer is subjected to the processes of photolithography mask patterning for front surface micromachining formation of flexible structures by successive etching of the silicon dioxide layer and the substrate in the hatched areas 25 to a predeterminded depth $a_z$.

During patterning simultaneously the structures of the micromechanical elements and functionalization element opening 10 shown in Fig.11 are obtained. This may be only the functionalization element opening 10, as in the example shown in Fig.5, or one of the functionalization elements as in the embodiments shown in Figures 7A, 8A, 8B and 9A.

When manufacturing sensors with functionalization element openings 10, the etching process is set and carried out at surface micromachining so that the sidewalls are as perpendicular to the surface of the wafer as possible, because the slope of the walls of the opening subsequently determines the inclination of the heterogeneous probe element assembled in it.

In a particularly preferred alternative embodiment of the method of the invention, unlike in hitherto known and widespread methods, where it is intended to use probe elements located in a plane parallel to the substrate plane, initially on the single-crystal silicon wafer V-shape trench functionalization elements or composed trenches with
suitable dimensions and orientation are formed on selected areas of the surface of the substrate. This is accomplished, as shown in Fig.12, by growing and depositing additional sacrificial and/or functionalization layers on the prepared in advance single-crystal silicon substrate 18, and this substrate is subjected to high temperature oxidation of both sides, which can be done by any known to those, skilled in the art, process. The thickness of the silicon dioxide layer 19 is such that it serves as a mask for the next bulk and surface micromachining of the silicon according to the description of the method of the invention below.

In a preferred embodiment of the method for obtaining oriented at will heterogeneous probe elements located on the surface of the sensor microcantilever as shown in Fig.12A, on the silicon dioxide layer 19 on front of the substrate, through an appropriate known in the field, physical or chemical process, two thin layers 20 and 20' are deposited to form therein a composed trench, although in some cases it is desirable that these layers be more than two. The thin layers 20 and 20' in this case are titanium with a thickness of 20 nm and gold with a thickness of 200 ran, and on top of the layers photoresistive layer is coated. Once this is done, a properly oriented photolithographic mask is patterned, and a window with a desired rectangular shape and orientation is obtained in the photoresist. Subsequently, though the mask controllable successive etching of the obtained layers is implemented in order to produce a desired cross section profile of the functionalization elements, thus forming a composed trench 21 with a width g in the layers 20 and 20' as shown in Fig.12A. Moreover, each top layer is a mask for etching the next layer located beneath, thus producing the desired form of the composed trench. The thickness of the layers, the amount g of composed trench 21 and its orientation towards other micromechanical elements of the sensor is determined by the dimension of the heterogeneous probe elements that will be used in the particular sensor, according to its application as well as to other requirements specific to it. These include requirements for wetting the material surface when fixing the heterogeneous element for the respective maximum temperature acceptable, known to those, skilled in the art.
The orientation of this structure towards the crystallographic directions of the silicon substrate \(18\) is at will.

In a preferred variant of the processes of functionalization elements trenches formation as shown in Fig.12B, when it is desired to obtain V-shaped trenches \(7\) on a single-crystal silicon substrate with a deposited oxide layer \(19\), a photoresist is used as mask for etching as well. After that a photolithographic mask with a desired orientation is patterned, while the rectangular windows from which functionalization elements will be produced are oriented in the \(<110>\) direction of the single-crystal silicon substrate. Then the silicon dioxide layer \(19\) is etched through this mask, and a rectangular window \(22\) on the dioxide layer with a size \(a\) and orientation in the \(<110>\) directions of the single-crystal silicon substrate is produced, as shown in Fig.12B. The resulting patterned silicon dioxide layer is a mask for the subsequent etching of the trench \(7\) in the silicon substrate up to a preset depth. Moreover, according to this preferred embodiment, the obtained functionalization elements are self-constraint V-shaped trenches. The production process proceeds with self-limitation, when a wet anisotropic etching of a (100) orientated single-crystal silicon substrate in potassium hydroxide (KOH) is performed. For this trench it is typical that its walls have the crystallographic orientation \(<111>\), its bottom is parallel to the surface of the substrate, and its depth \(d_i\) is determined by the photolithographic mask size \(a\) as it is set during the photolithographic patterning.

In another preferred embodiment of the first stage of the method according to the invention, as shown in Fig.12C-I and Fig.12C-II, when the desired sensor is with heterogeneous probe elements disposed in direction at will, in a plane parallel to and below the surface of the wafer, wherein the functionalization element is a composed trench, on the prepared single-crystal silicon substrate with grown silicon dioxide layer a photoresistive etching mask is coated. Similarly to the previous variant, then photolithography patterning of a mask for etching with a rectangular window with size \(b\) and with orientation at will is performed, so that the patterns to be oriented in the desired sensor application direction, as shown in Fig. 12C-I. The size \(b\) is determined mainly by
the diameter of the desired probe element to be assembled in the functionalization element. Then the silicon dioxide layer 19 is etched through the mask wherein the size of the mask b is transferred on the patterned silicon dioxide layer, thus forming a window 23, and the silicon dioxide is used as a mask for subsequent etching of a trench in the single-crystal silicon substrate. Then the silicon substrate 18 is isotropically etched through the resulting patterned mask layer of silicon dioxide 19 with an opening of width b, respectively wet or dry, until the area 24 is obtained, with further extending of this area up to the size b', as shown in Fig. 12C-II.

Then through the same mask of silicon dioxide a subsequent anisotropic dry etching is carried out, in which the window 23 with size b is projected onto the bottom of the area 24, thereby forming a secondary trench 23', which is part of the bottom of a composed trench as shown in the enlarged detail in Fig.12C-II. The second etching is carried out at low pressure, like reactive ion etching (RIE).

For the functionalization element composed trench 9, obtained by the mask with opening 23, it is significant that its axis is oriented at will, its depth d, is determined by the characteristics of probe element 4 and the sensor function, and its bottom is parallel to the surface of the substrate.

Further on the essential for the method of the invention alignment process is performed, wherein the photolithographical pattern is aligned with already existed functionalization elements, so that functionalization elements cross one-sidedly the surrounding sidewalls of the extended parts of the probe portion 6.

When manufacturing of SPM sensors with piezoresistive detection is desired, and accordingly appropriate n-type doped wafers are used, in which highly-doped surface areas and piezoresistive areas on the front surface and/or in sidewalls of trenches being in galvanic contact are previously obtained and where the photolithographic etching masks are allocated upon the surface of the wafer, for all method embodiments further aligning of these masks to the previously obtained piezoresistive and high-doped areas on the silicon wafer is performed.
In Fig. 13A the structure obtained after the processes of front surface micromachining in which individual elements are shown, like body 1 of the sensor, microcantilever 2, probe portion 6 and functionalization element 10 are formed simultaneously by anisotropic etching in the hatched areas 25 to a preset depth $a_e$ through a photolithographically defined mask, aligned to the previously obtained trench functionalization elements 7 or 9.

In the embodiments of the method for manufacturing sensors with piezoresistive detection, similar to the sensor shown in Fig. 10A, additionally alignment of the micromechanical elements microcantilever 2 with connecting part 5 and probe member 3 towards the highly-doped surface areas 13 and the areas with piezoresistive properties 14, as shown in Fig. 13B is provided.

Since the alignment between the two photolithographically patterned layers, which form the common flexible structures with embedded in sidewalls piezoresistors permit a wide tolerance, the resulting sensors are with reproducible parameters.

For those skilled in the art it is clear that these processes can be realized in different ways, with the method of the invention it is preferred the use of common positive tone photoresist applied by conventional spin coating method. The shape of the specifically formed flat flexible elements obtained at this stage are not limited.

An axonometric view of the functionalization elements obtained after etching at depth $d_f$ in form of V-shaped 7 or composed 9 trenches on flat flexible micromechanical structures are shown in Fig. 14A, with enlarged views of both kinds of trenches in Fig. 14B and Fig. 14C. As was already mentioned, when the axis of the trench is in the $<110>$ crystallographic direction, that trench is V-shaped, while in all other cases it is composed.

The view of the sidewall 8 of the probe portion 6 crossed by the V-shape trench functionalization element 7 is shown in Fig. 14B. An essential feature of the method of the invention is that the depth $d_f$ of the V-shaped trench and the position of the probe element is determined by the width $a$. In particular, where the width of the silicon dioxide
area preset by the of photolithographic patterning is of size \( a \) and the thickness of the microcantilever \( a_z \) is such, that is valid the equation

\[
a_z = a \cdot \tan 54.7°
\]  \hspace{1cm} (14)

than in this case the depth \( d_i \) of trench 7 is equal to a half of the thickness of the probe portion \( a_z \).

As a result, the method of this invention created unexpectedly the opportunity of manufacturing of trenches with reproducible parameters, including with a horizontal bottom located at preset distance from the surface of the substrate, in the middle along the thickness \( a_z \) of the probe portion 6.

The view of the crossed by the composed trench functionalization element 9, surrounding sidewall 8 of the probe portion 6 is shown in Fig.14C. An essential feature of a structure obtained according to the method of the invention is that its orientation can be at will. In addition, a common flat structure of the probe portion 6 with the composed trench functionalization element 9 is obtained. This conjoined structure enables the attachment of various heterogeneous probe elements allocated in different directions having different functional characteristics.

In a preferred embodiment of the method of the invention where the sensor contains both functionalization elements V-shaped trench 7 or composed trench 9 and an opening 10 in the shape of a right triangular prism, as the sensors shown in Fig. 7A, Fig. 8A, Fig. 8B and Fig. 9A having, the functionalization element opening 10 is aligned to the functionalization elements trenches so that in the opening 10 the imaginary intersection point A of the axes of the functionalization elements trenches to be located, which trenches are already formed on the processed single-crystal silicon structure, while the subsequent etching of the silicon dioxide layer and the substrate is performed to the preset depth \( a_z \). Further, the resulting flexible structure is treated similarly to the other sensors, object of the present invention.

When manufacturing of SPM sensors with piezoresistive detection is desired, after executing the processes described so far, further metallization patterning processes are
performed, wherein in contact with the high conductivity areas on the probe member, the microcantilever and the sensor body connecting metal paths with contact pads are obtained. These metallization processes are performed by methods known to those, skilled in the art.

5 **Bulk micromachining of flexible micromechanical structures**

The so processed single-crystal silicon structure, is subjected to photolithographical patterning processes of the silicon dioxide layer on the back surface of the silicon wafer, subsequent bulk etching of the back side of silicon wafer up to a predetermined thickness of the residual silicon layer and release of the flexible micromechanical elements, which can be achieved in any known in the field manner. Moreover, during the bulk micromachining a planar carrier structure is obtained which connects laterally and holds together the individual sensors, which are usually obtained simultaneously on a substrate within a single technology process chain.

10 **Heterogeneous probe elements’ assembly**

Into the functionalization elements of the flexible micromechanical elements produced during the previous stage, heterogeneous probe elements are then positioned and fixed to the bottom of functionalization elements.

The positioning of probe elements in the functionalization elements is accomplished by various methods known to those, skilled in the art. Fig.15A shows a particularly preferred method for the production of a sensor with an elongated heterogeneous probe element, using V-shaped trench functionalization element 7. An elongated heterogeneous probe element 4 with a cylindrical shape and a small diameter is positioned in the trench, in this case a CNT or a bundle of such CNTs. Other elements can be used as well, such as boron or boron-nitride nanotubes, nano-fibres, nano-wires micro-/nanocrystals, or elements of complex configuration, having an elongated cylindrical part. Since the diameter of the CNT is smaller than the width of the trench, typically several hundred to one thousand times smaller, CNTs are positioned on the bottom of the trench or falls by itself to bottom thus self-positioned, as shown in Fig.15B.
In the particularly preferred embodiment with self-positioning it is sufficient before placing the CNT probe element, to position it relative to the axis of the trench with an accuracy equal to one half of the dimension $a$.

The positioning of an elongated heterogeneous cylindrical probe element is carried out in similar way, when the functionalization element formed on the flexible micromechanical element is a composed trench 9. When self-positioning is used it is sufficient before placement, the probe element to be positioned relative to the axis of the trench with an accuracy equal to one half the size $b'$ of the composed trench.

The positioning of heterogeneous probe elements in functionalization elements openings 10 is accomplished with the sensor initially being oriented vertically, so that the inner edge of the prism opening containing point A, through which the probe element will pass, to be in the lowest position relative to the surrounding opening sidewalls. Then the probe element is put into the opening with a micromanipulator and is positioned or self-positioned towards the lowest inner edge. When self-positioning is used it is sufficient before placing, the CNT to be positioned relatively to opening with an accuracy, equal to one half the size of the side of the triangular prism.

After placing the elongated cylindrical heterogeneous probe elements, they are fixed to the bottom of the trench through the application of a suitable coating 26 or without it. It is necessary that the coating 26 has good adhesion to the material of the functionalization elements - trenches 7, 9 or opening 10 as shown in Fig. 16A.

When the heterogeneous probe element 4 is positioned along the axis of the functionalization element 7, 9 or 10, the length $l_p$ of the part of probe element 4, which protrudes beyond the probe portion, is set in advance.

In a preferred embodiment of the method of the invention shown in Fig. 16B, coating 26 is applied to trench 7, of over the cylindrical probe element 4, positioned and placed in advance on the bottom of said trench. Depending on the ease of implementation, fixation of the probe elements can be accomplished by various methods as required and with the appropriate amount of material for each probe element.
It is proceeded in similar way when the other preferred embodiment of the method is carried out and CNT, CNT bundles or other probe elements 4 with appropriate properties are fixed by application of a suitable coating 26, which has good adhesion to the material of composed trench 9, as shown in Fig. 16C. Depending on the ease of implementation, fixation of probe elements can be accomplished by various methods as required using the appropriate amount of coating material 26 for each probe element.

Probe elements can be attached to the functionalization elements including known from state of the art methods of fixation by local e-beam welding in a scanning electron microscope chamber, as well as any other known and feasible method. These alternatives are used particularly to accomplish the attachment of probe element 4' in openings 10, shaped like a right triangular prism.

In the manner described herein probe elements or bundles of such elements having properties suitable for the particular application can be placed and fixed independently at will. At the same time, in microcantilever structures with aligned functionalization elements different probe elements can be assembled.

Finally, finished sensors are separated from their supporting planar carrier structure, which can be accomplished using various methods as known in the field.
CLAIMS

1. A sensor for scanning probe microscopy, which includes formed jointly out of a single crystal substrate a body, a protruding out of it microcantilever and a protruding out of the microcantilever free end probe member, characterized in that said probe member is flat and is located in one plane with the body and the microcantilever, representing their common flat surface; with the probe member comprising a probe portion with formed on it at least one concavely shaped functionalization element, within which an elongated heterogeneous probe element, whose axis is parallel to the common flat surface or is perpendicular to it, is assembled; whereas on the microcantilever and/or on the probe member for each $X, Y$ and/or $Z$ measurement direction with said sensor, an area is individualized with determined geometric dimensions, which define the individual bending oscillation characteristics of said microcantilever and/or probe member in the relevant measurement direction.

2. A sensor for scanning probe microscopy according to the preceding claim, characterized in that the concavely shaped functionalization element is a trench with a depth less than the thickness of the probe portion, or an opening with a depth equal to the thickness of the probe portion; whereas the form of the trench is V-shaped or composite, and said trench is with a constant cross section along its length and is located in parallel to the plane of the common flat surface; whereas the opening is of a triangular prism shape and is formed perpendicularly to the plane of the common flat surface; whereas every concavely shaped functionalization element has a slope of the walls and cross section dimension sufficient to provide positioning of the probe element, which is assembled in said functionalization element.

3. A sensor for scanning probe microscopy according to claim 2, characterized in that the geometric dimensions of the individualized areas of microcantilever and/or probe member determining individual resonance frequencies for each measurement direction $X, Y$ and/or $Z$, for every two directions of measurement are defined in accordance with the relations for discernibleness of the resonance frequencies:

$$Q' (f_{oj} - f_{oj+i}) \geq f_{oj}$$ and

$$f_{oj} / f_{oj+i} = a_{oj} / a_{oj+i},$$
wherein $\gamma$ is a coefficient in the range from 0.1 to 10, preferably from 0.2 to 1.0, characterizing the method selected to discern the measurement direction depending on the frequency response when bending the flexible micromechanical element;

$f_{0j}$ is the resonance frequency of the microcantilever or probe member upon bending in the direction $j$;

$f_{0j+i}$ is the resonance frequency of the microcantilever or probe member upon bending in the direction $j+i$ respectively;

$Q$ is the average value of the quality factors of the microcantilever and/or probe member for the measurement directions $j$ and $j+i$;

$a_j$ and $a_{j+i}$ are determined geometrical dimensions, correspondingly for the microcantilever and/or the probe member.

4. A sensor for scanning probe microscopy according to claim 2, characterized in that on the probe portion two functionalization elements are formed, the axes of which make an angle less than 180° and have an imaginary intersection point, which falls outside the probe portion in the measurement direction.

5. A sensor for scanning probe microscopy according to claim 3, characterized in that the probe member is provided with connecting part which conjoins it with the microcantilever, and in its probe portion there is at least one additional laterally extended region with a functionalization element, in which an elongated heterogeneous probe element is assembled respectively; whereas the axes of such two functionalization elements make an angle of less than 180° and have an imaginary intersection point falling in the central area of the probe member.

6. A sensor for scanning probe microscopy according to claim 5, characterized in that the additional laterally extended regions with formed on them functionalization elements are two, with assembled in them elongated heterogeneous elements so that the axes of said three functionalization elements have a common imaginary intersection point falling in the central area of the probe portion, and each one of the elongated heterogeneous probe elements makes with the microcantilever axis an angle in the range between 0° and 180°.
7. A sensor for scanning probe microscopy according to claim 6, characterized in
that in the central area of the probe portion a functionalization element opening is
formed, in which the imaginary intersection point of the axes of the remaining
functionalization elements fall; while the axis of the elongated heterogeneous probe
element assembled in the opening and protruding at least one-sidedly thereof, passes
through the imaginary intersection point of the axes of the remaining functionalization
elements.

8. A sensor for scanning probe microscopy according to any of the preceding claims,
characterized in that it has from one to four, optionally identical or different, elongated
heterogeneous probe elements with a longitudinal axis of symmetry and an aspect ratio
from 1:10 to 1:5.10^3; selected among carbon, boron, boron-nitride or metal nanotube,
nanofibre, nanowire, nanorod; or micro-, nano-crystalls; or probe elements constituted of
heterogeneous parts with spherical, pyramidal or multi-pyramidal form; or probe
elements additionally specifically functionalized; and preferably single nanotubes or a
bundle of uniform nanotubes, each representing single wall or multiwall carbon
nanotube, including electrically conductive nanotubes; boron, or boron-nitride nanotubes.

9. A sensor for scanning probe microscopy according to any of the preceding claims,
characterized in that the areas with determined geometric dimensions contain
additionally: piezoresistors transducing the displacement of the microcantilever and/or
probe member in any measurement direction in an electrical signal; which are disposed in
the plane of the common flat surface and/or planes perpendicular to it; and highly doped
areas through which piezoresistors are in galvanic contact with the metal paths formed on
the body, microcantilever and probe member, with the metal paths being provided with
contact pads to the system measuring the electrical parameters.

10. A method for three-dimensional measurement with feedback control with a sensor
for scanning probe microscopy of a sample surface, in which the sensor and/or
investigated sample are translated relative to one another by the scanning system to reach
any one of the points on the surface where a measurement is carried out, characterized
in that it is used a sensor with one microcantilever and one probe member with at least
one probe element having a common flat surface and individual oscillation characteristics
of bending without torsion in each direction of measurement, which characteristics are
discernible from one another upon measurement and the number of the probe elements is sufficient to ensure measurement in each of the directions X, Y and Z; whereas after reaching the corresponding point on the sample surface, where measurement is carried out, the sensor and the sample are set in a static position and the surface surrounding of the probe element apex is subjected to investigation stepwisely in each of the directions X, Y and Z without translating the sensor and/or sample through bringing the microcantilever and probe member into exited oscillation by periodic actuation, which oscillation is sequentially carried out with working amplitude magnitude and test amplitude magnitude for each of the directions X, Y and Z, and with a frequency in range containing the resonance frequency for the measured particular direction of measurement; and the data for sensor scanning system coordinates and oscillation amplitude magnitudes needed for keeping constant by feedback control the value of the physical quantity featuring interaction of oscillating probe element apex with the sample: oscillation phase, amplitude magnitude, resonance frequency or any other physical quantity are acquired and processed to obtain a corresponding three-dimensional characteristics of the sample.

A method for manufacturing a sensor for scanning probe microscopy as claimed in any of the claims 1 to 9, characterized in that on a p-type doped wafer of single crystal silicon with (100) orientation, with a specific resistivity of 0.01 to 20 Ω·cm or on a n-type doped wafer with a specific resistivity of 0.003 to 20 Ω·cm, in sequence the following processes are carried out: formation of at least one upper sacrificial layer, and after that front surface micromachining by patterned etching, optionally photolithographically aligned to formed in advance on the front wafer surface at least one concavely shaped functionalization element with a depth less than the thickness of the probe portion; thus forming simultaneously in a single plane the body of the sensor, the protruding out of it microcantilever, the probe member and optionally, a centrally located concavely shaped functionalization element with a depth equal to the thickness of the probe member, thus obtaining a body and a flat flexible structure with at least one concavely shaped functionalization element which are subjected to back side surface micromachining of the silicon wafer and to a subsequent bulk micromachining and releasing of the entire sensor structure; while simultaneously a planar carrier structure is formed; then in the created at
least one concavely shaped functionalization element an elongated heterogeneous probe element is assembled through positioning or self-positioning it at the bottom of the concavely shaped functionalization element and fixing to the bottom; and finally, the completed sensor is released from the carrying planar structure.

12. A method according to claim 11, characterized in that in which the formed on the surface of the wafer at least one concavely shaped functionalization element with depth less than the thickness of the probe portion, is a V-shaped or composed trench produced via photolithographic patterning and at least one subsequent etching process.

13. A method according to claim 12, characterized in that a n-type doped single crystal silicon wafer with a specific resistance from 1 to 20 Ω-cm is used, which additionally contains created on it in advance doped and galvanically connected highly conductive and piezoresistive areas, to which during the surface micromachining in the process of photolithography are aligned the areas with determined geometrical dimensions of the microcantilever and probe member; then through the front surface micromachining of those doped areas piezoresistors are formed; and after that metallization patterning processes are carried out in which, in contact with the highly conductive areas on the probe member, on the microcantilever and on the sensor body connecting metal paths with contact pads are produced.
INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

INV. G01Q60/38 G01Q70/10 G01Q70/12 G01Q10/06

ADD.

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

G01Q

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

<table>
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<th>Category</th>
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<th>Relevant to claim No.</th>
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<td>JP 6 307852 A (OLYMPUS OPTICAL CO) 4 November 1994 (1994-11-04)</td>
<td>1, 9, 11, 13</td>
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<td>Y</td>
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<td>EP 1 672 648 A1 (NANOWORLD AG [CH]) 21 June 2006 (2006-06-21)</td>
<td>1,8</td>
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<td>A</td>
<td>figure 5 paragraph [0018]</td>
<td>2-7,9-13</td>
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Further documents are listed in the continuation of Box C. See patent family annex.

* Special categories of cited documents:
  * "A" document defining the general state of the art which is not considered to be of particular relevance
  * "E" earlier document but published on or after the international filing date
  * "L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another document or other special reason (as specified)
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*A* document member of the same patent family

Date of the actual completion of the international search 11 March 2011

Date of mailing of the international search report 18/03/2011

Name and mailing address of the ISA:

European Patent Office, P.B. 5818 Patentlaan 2
NL-2280 HV Rijswijk
Tel. (+31-70) 340-2040,
Fax: (+31-70) 340-3016

Authorized officer

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INTERNATIONAL SEARCH REPORT

Box No. II  Observations where certain claims were found unsearchable (Continuation of item 2 of first sheet)

This international search report has not been established in respect of certain claims under Article 17(2)(a) for the following reasons:

1. ☐ Claims Nos.: because they relate to subject matter not required to be searched by this Authority, namely:

2. ☐ Claims Nos.: because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out, specifically:

3. ☐ Claims Nos.: because they are dependent claims and are not drafted in accordance with the second and third sentences of Rule 6.4(a).

Box No. III  Observations where unity of invention is lacking (Continuation of item 3 of first sheet)

This International Searching Authority found multiple inventions in this international application, as follows:

see additional sheet

1. ☒ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims.

2. ☐ As all searchable claims could be searched without effort justifying an additional fees, this Authority did not invite payment of additional fees.

3. ☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims for which fees were paid, specifically claims Nos.:

4. ☐ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claims Nos.:

Remark on Protest ☐ The additional search fees were accompanied by the applicant’s protest and, where applicable, the payment of a protest fee.

☒ The additional search fees were accompanied by the applicant’s protest but the applicable protest fee was not paid within the time limit specified in the invitation.

☒ No protest accompanied the payment of additional search fees.
This International Searching Authority found multiple (groups of) inventions in this international application, as follows:

1. claims: 1-9, 11-13

   SPM planar sensor consisting of a body, a cantilever and a free end probe member all in the same plane, wherein the free end probe member has a concave trench or opening for placing an elongated probe element, and method for its manufacture.

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2. claim: 10

   Method for measurement in three dimensions with a planar SPM probe, wherein at each point the three different resonance frequencies in the three different orthogonal directions are measured with two different oscillation amplitudes.

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<td>JP 6307852</td>
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