



(51) International Patent Classification:

G01Q 60/06 (2010.01) G01Q 60/34 (2010.01)
G01Q 60/18 (2010.01) G01N 21/3581 (2014.01)

(21) International Application Number:

PCT/US2024/010366

(22) International Filing Date:

04 January 2024 (04.01.2024)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

63/478,453 04 January 2023 (04.01.2023) US
63/478,448 04 January 2023 (04.01.2023) US

(71) Applicant: **BROWN UNIVERSITY** [US/US]; Box 1949,
Providence, RI 02912 (US).

(72) Inventors: **MITTLEMAN, Daniel**; 121 Benevolent Street,
Providence, RI 02906 (US). **PIZZUTO, Angela**; 503 River
View Ln, Norwich, CT 06360 (US).

(74) Agent: **HOLMANDER, Daniel, J.**; Adler Pollock & Shee-
han P.C., One Citizens Plaza, 8th Floor, Providence, RI
02903 (US).

(81) Designated States (unless otherwise indicated, for every
kind of national protection available): AE, AG, AL, AM,
AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ,
CA, CH, CL, CN, CO, CR, CU, CV, CZ, DE, DJ, DK, DM,
DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT,
HN, HR, HU, ID, IL, IN, IQ, IR, IS, IT, JM, JO, JP, KE, KG,
KH, KN, KP, KR, KW, KZ, LA, LC, LK, LR, LS, LU, LY,
MA, MD, MG, MK, MN, MU, MW, MX, MY, MZ, NA,
NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO,
RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, ST, SV, SY, TH,
TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, WS,
ZA, ZM, ZW.

(54) Title: SYSTEM AND METHOD FOR SCANNING NEAR-FIELD OPTICAL MICROSCOPY

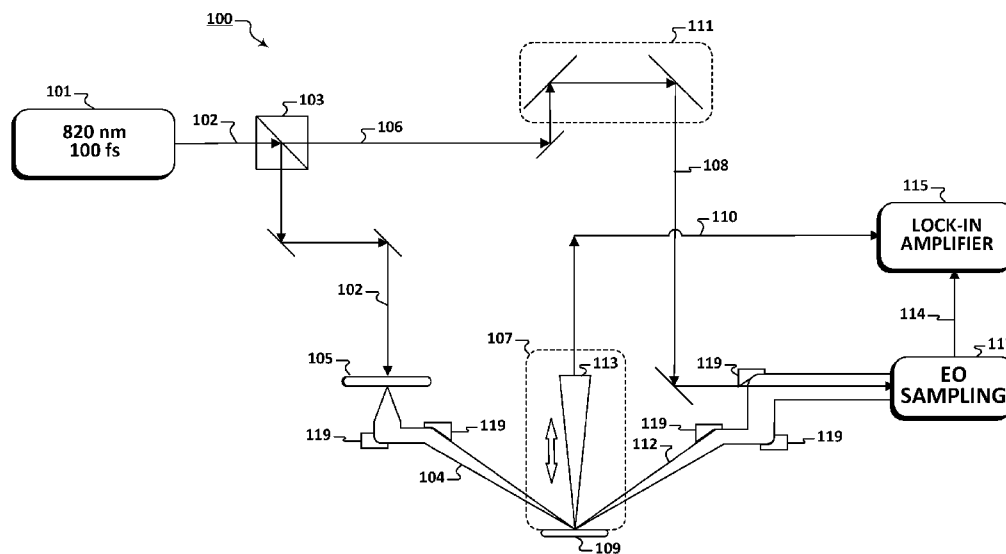


FIG. 1

(57) Abstract: A method for scanning near-field optical microscopy comprises illuminating an apertureless atomic force microscopy (AFM) probe with electromagnetic energy having a frequency in the Terahertz range, where the sample under observation includes a dielectric layer having a thickness greater than the radius of the tip of the AFM probe. A system for scanning near-field optical microscopy comprises a collimated light source for emitting collimated light, a photoconductive antenna for converting collimated light into electromagnetic energy having a frequency in the Terahertz range, an AFM probe, a sample comprising a dielectric layer, the dielectric layer having a thickness greater than the radius of the probe tip; and a detector configured to detect energy that has interacted with the sample.

(84) Designated States (*unless otherwise indicated, for every kind of regional protection available*): ARIPO (BW, CV, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SC, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, ME, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

Published:

— *with international search report (Art. 21(3))*

SYSTEM AND METHOD FOR SCANNING NEAR-FIELD OPTICAL MICROSCOPY

CROSS-REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority to U.S. Provisional App. No. 63/478,448 filed January 4, 2023, and to U.S. Provisional App. No. 63/478,453 filed January 4, 2023.

BACKGROUND

Field

[0002] The present disclosure relates generally to scanning near-field optical microscopy, and, in particular, to apertureless scanning near-field optical microscopy, and, further, to terahertz near-field optical microscopy.

Description of the Problem and Related Art

[0003] The implementation of near-field studies in the terahertz spectral range has been an active area of research. Although these aperture-based techniques have become quite sophisticated, they are ultimately limited by the physics of the transmission of radiation through sub-wavelength apertures, which becomes resolving sub-micron-scale features with terahertz waves (with wavelengths of hundreds of microns or more; 1 THz $\sim$ 300 μ m).

[0004] A significant breakthrough occurred with the introduction of scattering-type apertureless scanning near-field optical microscopy (s-SNOM) [2]. This technique relies on the scattering of light from a sub-wavelength-sized metal tip inside an atomic force microscope (AFM), which is oscillating (tapping) near a sample surface (but not touching it). The radiation scattered from the tip-sample system contains information about the dielectric properties of the nearby surface. The technique can therefore be used for spectroscopy. Moreover, since the scattered light intensity is a nonlinear function of tip-sample separation, one can filter the signal at a harmonic of the tip's tapping frequency, such that the extracted spectroscopic information is characteristic of only the tiny region of the sample directly underneath the tip. This enables spectroscopic imaging with a spatial resolution determined

by the size of the metal tip, essentially independent of the wavelength of the incident radiation.

[0005] Typical of s-SNOM is the use of an Atomic Force Microscope (AFM) which is a high-resolution type of scanning probe microscope that provides three-dimensional surface topography images at the nanoscale. The AFM operates by scanning a sharp tip over the surface of a sample while measuring the interaction forces between the tip and the atoms on the surface. The heart of the AFM is a tiny cantilever with a sharp tip at its free end. The tip is typically made of a material such as silicon or silicon nitride and has a radius of a few nanometers. The AFM tip is brought very close to the sample surface, within a few nanometers. As the tip scans across the surface, the interaction forces (such as van der Waals forces, electrostatic forces, and chemical forces) between the tip and the sample cause the cantilever to bend. A laser beam is typically focused on the back of the cantilever. The deflection of the cantilever causes changes in the position of the laser beam on a position-sensitive photodetector. These changes are then used to generate a feedback signal to maintain a constant force between the tip and the sample, allowing precise control of the tip-sample distance. The feedback mechanism is used to maintain a constant force as the tip scans the sample in a raster pattern. The vertical movements of the tip are recorded, and this information is used to construct a topographic image of the sample surface with nanometer-scale resolution. AFMs can operate in different modes, including contact mode, tapping mode, and non-contact mode, each offering specific advantages and applications. s-SNOM, therefore, combines the capabilities of Atomic Force Microscopy (AFM) with near-field optical microscopy to achieve high-resolution imaging beyond the diffraction limit of conventional optical microscopes.

[0006] The AFM includes a sharp tip at the end of a cantilever, which is used for topographic imaging of the sample surface. The AFM tip is brought into proximity with the sample surface, typically within a few nanometers. As the AFM tip scans the sample surface, it detects the topography with high spatial resolution, providing topographic information. Simultaneously, the sample is illuminated with an optical source, often a laser, which is focused on the AFM tip. The near-field interaction between the sample and the AFM tip modifies the optical signals, and the scattering of light is influenced by the local properties of the sample. The scattered optical signals, which carry information about the near-field optical properties of the sample, are collected.

[0007] The AFM and optical signals are detected simultaneously, allowing for the correlation of topographic information obtained by the AFM with the near-field optical properties of the sample. The combination of AFM and near-field optics allows for sub-wavelength spatial resolution, enabling researchers to obtain detailed images of nanoscale features and optical properties of the sample.

[0008] However, few studies have considered the possibility of imaging sub-surface features, situated some distance beneath a transparent (to THz waves) capping layer that is thicker than the size of the field confinement region near the AFM tip, which is essentially equal to the tip radius).

SUMMARY

[0009] For purposes of summary, certain aspects, advantages, and novel features are described herein. It is to be understood that not necessarily all such advantages may be achieved in accordance with any one particular embodiment. Thus, the apparatuses or methods claimed may be embodied or carried out in a manner that achieves or optimizes one advantage or group of advantages as taught herein without necessarily achieving other advantages as may be taught or suggested herein.

[0010] In a first aspect, a method for imaging metallic patterns deposited on the surface of semiconductor materials comprises illuminating an apertureless atomic force microscopy (AFM) probe with electromagnetic energy having a frequency in the Terahertz range, where the sample under observation comprises a dielectric layer having a thickness greater than the radius of the tip of the AFM probe.

[0011] In another aspect, a method is disclosed for non-destructive testing using an AFM probe where the method comprises detecting terahertz electromagnetic energy reflected from a sample, the terahertz electromagnetic energy originating from a collimated light source, and the sample includes a dielectric layer with a thickness greater than the radius of the probe tip, and where the probe tip oscillates at a distance of between about 5 nm and about 255 nm from said sample.

[0012] In yet another aspect, an exemplary system for scanning near-field optical microscopy comprises a collimated light source for emitting collimated light, a photoconductive antenna for converting collimated light into electromagnetic energy having a frequency in the Terahertz range, an AFM probe, a sample comprising a dielectric layer, the dielectric layer having a thickness greater than the radius of the probe tip; and a detector configured to detect energy that has interacted with the sample.

BRIEF DESCRIPTION OF THE DRAWINGS

[0013] The disclosed system and method are described with reference to the accompanying drawings. In the drawings, like reference numbers indicate identical or functionally similar elements. Additionally, the left-most digit(s) of a reference number identifies the drawing in which the reference number first appears.

[0014] FIG. 1 is a diagram of an exemplary system for scanning near-field optical microscopy (SNOM);

[0015] FIG. 2 is an isolated view of showing relationship between an AFM probe tip and a sample;

[0016] FIG. 3A is a scanning electron microscope image of a sample;

[0017] FIG. 3B is an AFM image of the sample of FIG. 3A; and

[0018] FIGs. 4A through 4D are s-SNOM images using the method disclosed herein.

DETAILED DESCRIPTION

[0019] The various embodiments of the system and method disclosed herein and their advantages are best understood by referring to FIGs. 1 through 4D of the drawings. The elements of the drawings are not necessarily to scale, emphasis instead being placed upon clearly illustrating the novel features and principles of operation. Throughout the drawings, like numerals are used for like and corresponding parts of the various drawings.

[0020] Furthermore, reference in the specification to “an embodiment,” “one embodiment,” “various embodiments,” or any variant thereof means that a particular feature or aspect described in conjunction with the particular embodiment is included in at least one embodiment. Thus, the appearance of the phrases “in one embodiment,” “in another embodiment,” or variations thereof in various places throughout the specification are not necessarily all referring to its respective embodiment.

[0021] With reference to FIG. 1, an exemplary system 100 for scanning near-field optical microscopy (SNOM) comprises an atomic force microscope (AFM) 107 that includes a cantilevered, apertureless probe 113. A light source 101 emits pulsed, collimated light 102, preferably at a wavelength of 820 nm and a pulse width of approximately 100 fs. Pulsed light 102 interacts with beam-splitter 103 which redirects a portion of pulsed light 102 to a photoconductive antenna (PCA) 105 which converts pulsed light 102 into radio frequency (RF) energy 104 in the terahertz frequency range. Terahertz RF energy 104 is directed via waveguides 119 to probe 113 and sample 109 under observation in AFM 107. A portion of pulsed light emerges from beam splitter 103 to provide a reference signal 106 which is directed to a delay line 111.

[0022] FIG. 2 shows probe 113 in isolation above sample 109 separated by a gap 202 from probe tip 205 to the surface of sample 109. In operation, probe 113 is oscillated 206 at a tapping frequency such that the distance of gap 202 varies between about 5 nm and about 255 nm. Tapping frequency may be about 20 Hz. Sample 109 may comprise substrate 203 which may include defects in its surface and a dielectric capping layer 201 overlaying substrate 203, which may be, for example, a silicon wafer. Dielectric layer 201 may comprise, for example, a silica layer and may be a thickness 204 that in some embodiments is greater than the radius 208 of probe tip 205. In another embodiment, thickness 204 is about five times greater than the radius 208 of probe tip 205. In yet another embodiment, thickness 204 of layer 201 is between about 200 nm to about 400 nm.

[0023] For typical AFM, the tip is indeed made of silicon or silicon nitride, a dielectric, and the apex can be as small as a few nanometers in diameter. But for this terahertz application, “typical” AFM tips cannot be used. This method must employ metal tips. Any metal tip may be used, but in a preferred embodiment, tips may be composed of a platinum-iridium alloy. Also, the conventional AFM tip is pyramidal shaped, but this places the

cantilever arm too close to the sample surface such that the terahertz beam is blocked from reaching the surface by the cantilever itself. Therefore, the conical tip needs to be at the end of a relatively long (like 80 micron) cylindrical shank, so that the cantilever which holds it is farther away from the sample surface. In this system 100, probe 113 may have between about 80 to about 100 micron shanks, and with that taper down to a tip of about 20 nanometers minimum size.

[0024] Referring again to FIG. 1, terahertz energy 104 is directed to probe 113, and in particular, to probe tip (FIG. 2: 205) whereupon energy 104 is scattered toward sample 109. Reflected energy 112 is collected by waveguides 119 and directed to electro-optical sampler 117 for detection and analysis. At the same time a portion of collimated light exits beam-splitter 103 as reference signal 106 which is directed to a delay line 111 providing an optical signal 108 to electro-optical sampler 117 where it is modulated by reflected energy 112. A modulated detected signal 114 is conveyed to lock-in amplifier 115.

[0025] AFM 107 also provides a demodulation signal 110 which is the reference for the lock-in amplifier 115. Demodulation signal 110 includes data regarding the tapping frequency of the AFM 107 cantilevered probe 113, or a multiple of it. AFM 107 supplies a signal which is the position of the cantilevered probe 113 vs. time, and which therefore oscillates at the mechanical resonance of the cantilever, which is approximately 20 kHz in this case. Demodulation signal 110 can also provide signals at multiples of this frequency (i.e., approximately 40 kHz, 60 kHz, etc), which are referred to as the “harmonics” of the tapping frequency. Lock-in amplifier 115 demodulates detected signal 114 at one of these harmonic frequencies, by providing this as the reference frequency for the lock-in. Most often the 2nd harmonic which is near 40 kHz is used. Exact values of these frequencies are determined by the precise mass of the cantilever, and are not fundamental to the method.

[0026] A program of experimental study was conducted of well characterized and controlled samples consisting of transistor devices fabricated on one side of a thinned silicon wafer (with various different thicknesses), together with a computational effort directed towards the development of a numerical model for quantitative extraction of material parameters of the buried structures. The possibility was explored that the incident THz field, plasmonically enhanced through near-field interactions with the extended metal AFM tip,

may be large enough to perturb the local carrier density in the semiconductor underneath the tip, leading to a measurable effect on the electrical properties of the nearby silicon device.

[0027] The feasibility of obtaining near-field spectroscopic images through a low-loss high-dielectric layer was investigated. A typical sample consisted of a series of well-spaced transistor structures, with known spatially varying doping profiles, fabricated on one side of a high-resistivity Si wafer which is then subsequently thinned to a desired thickness. Samples were probed using s-SNOM methods from the thinned back side of the wafer. The strength of the scattered THz near-field signal and the lateral spatial resolution, as a function of the thickness of this dielectric layer, over a range of thicknesses from 0.1-1 micron were determined. When possible, images of the transistor structures were formed using both of these measurement parameters, employing the 2D scanning capabilities of the AFM stage. Spectroscopic information was investigated in an attempt to extract quantitative measures of local doping density at each pixel of these images.

[0028] Fig. 3A shows a scanning electron microscope (SEM) image of a portion of one such sample. FIG. 3B is an atomic force microscope (AFM) topography image of a small portion indicated by the box shown. The topography highlights three metal vias 301a-c protruding from the SiO₂ surface, with an average distance above the (otherwise featureless) background of roughly 45 nanometers.

[0029] The corresponding THz reflection images, for four different harmonic demodulation orders, are shown in Fig. 4A – 4D. Here, one can clearly see three underlying vertical metal lines 401a-c which are only faintly visible in the SEM image of FIG. 3A, and do not appear at all in the AFM topography of FIG. 3B, because they are buried under a silica cap layer. Subsequent analysis of this sample indicated that this cap layer is roughly 200 nm thick, about a factor of 5 larger than the radius of the AFM tip. This key experimental result establishes that the apertureless THz near-field imaging technique can indeed be used to study buried features, while preserving the nanoscale spatial resolution, despite the conventional wisdom that the confined near field extends only about one tip diameter away from the end of the AFM tip.

[0030] As described above and shown in the associated drawings, the present invention comprises a system and method for scanning near-field optical microscopy. While

particular embodiments have been described, it will be understood, however, that any invention appertaining to the system and method described is not limited thereto, since modifications may be made by those skilled in the art, particularly in light of the foregoing teachings. It is, therefore, contemplated by the appended claims to cover any such modifications that incorporate those features or those improvements that embody the spirit and scope of the invention.

WHAT IS CLAIMED IS:

1. A method for imaging metallic patterns deposited on a surface of semiconductor materials, said method comprising:
 - illuminating an apertureless atomic force microscopy (AFM) probe with electromagnetic energy having a frequency in a Terahertz range, said probe having a probe tip, said probe tip having a radius, wherein said electromagnetic radiation is redirected onto a sample, said sample comprising a dielectric layer having a thickness greater than said radius; and
 - detecting said electromagnetic energy that has interacted with said sample.
2. The method of Claim 1, wherein said thickness of said dielectric layer is at least five times greater than said radius.
3. The method of Claim 2, wherein said thickness is about 200 nm to about 400 nm.
4. The method of Claim 1, further comprising oscillating said probe tip such that said probe tip is between about 5nm and about 255 nm from said sample.
5. The method of Claim 4, wherein said thickness of said dielectric layer is at least five times greater than said radius.
6. The method of Claim 5, wherein said thickness is about 200 nm to about 400 nm.
7. A method for non-destructive testing using an AFM probe, the probe having a probe tip, the probe tip having a radius, the method comprising the steps of:
 - detecting terahertz electromagnetic energy reflected from a sample, said terahertz electromagnetic energy originating from a collimated light source, said sample having a dielectric layer with a thickness greater than said radius, and wherein said probe tip oscillates at a distance of between about 5 nm and about 255 nm from said sample.
8. The method of Claim 7, wherein said thickness is about 200 nm to about 400 nm.

9. A system for scanning near-field optical microscopy comprising:
 - a collimated light source for emitting collimated light;
 - a photoconductive antenna for converting said collimated light into electromagnetic energy having a frequency in a terahertz range;
 - an AFM probe, said probe having a probe radius;
 - a sample comprising a dielectric layer, said dielectric layer having a thickness greater than said probe radius; and
 - a detector configured to detect energy that has interacted with said sample.
10. The system of Claim 9, wherein said thickness is about 200 nm to about 400 nm.
11. The system of Claim 9, wherein said probe is configured to oscillate at a distance of between about 5 nm and about 255 nm from said sample.
12. The system of Claim 11, wherein said thickness is about 200 nm to about 400 nm.
13. The system of Claim 9, wherein said thickness of said dielectric layer is at least five times greater than said radius.
14. The system of Claim 9, further comprising a demodulator for demodulating a detected signal received from said detector.
15. The system of Claim 14, wherein said thickness is about 200 nm to about 400 nm.
16. The system of Claim 15, wherein said probe is configured to oscillate at a distance of between about 5 nm and about 255 nm from said sample.

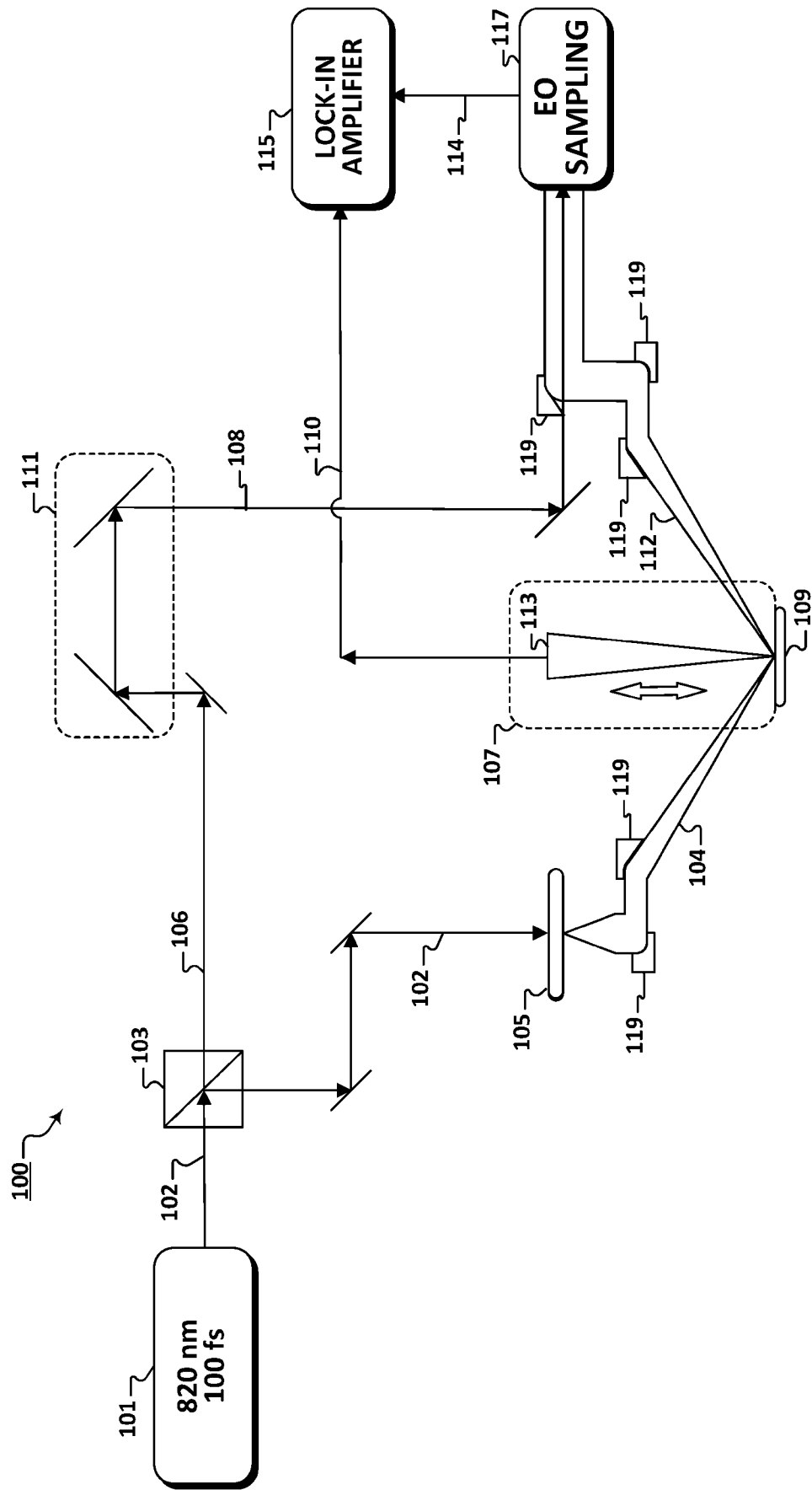


FIG. 1

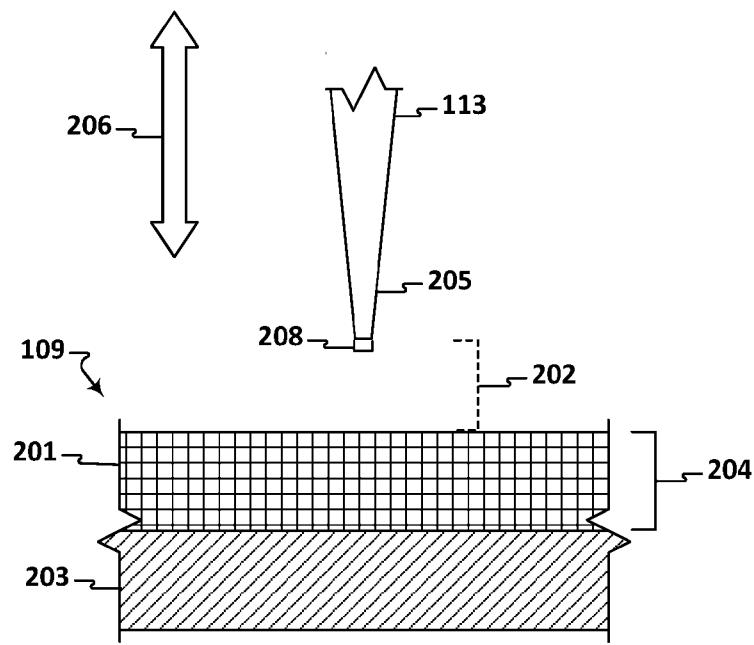


FIG. 2

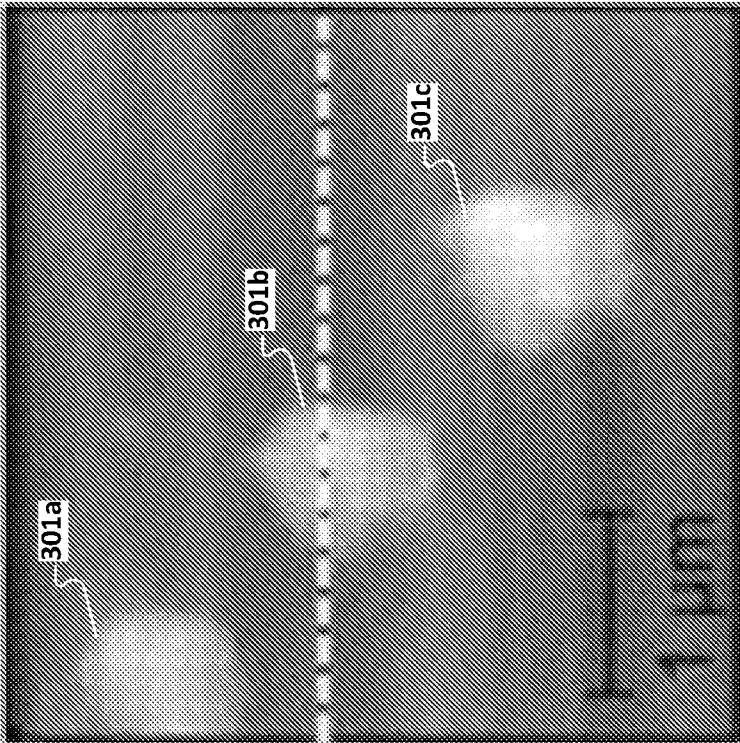


FIG. 3B

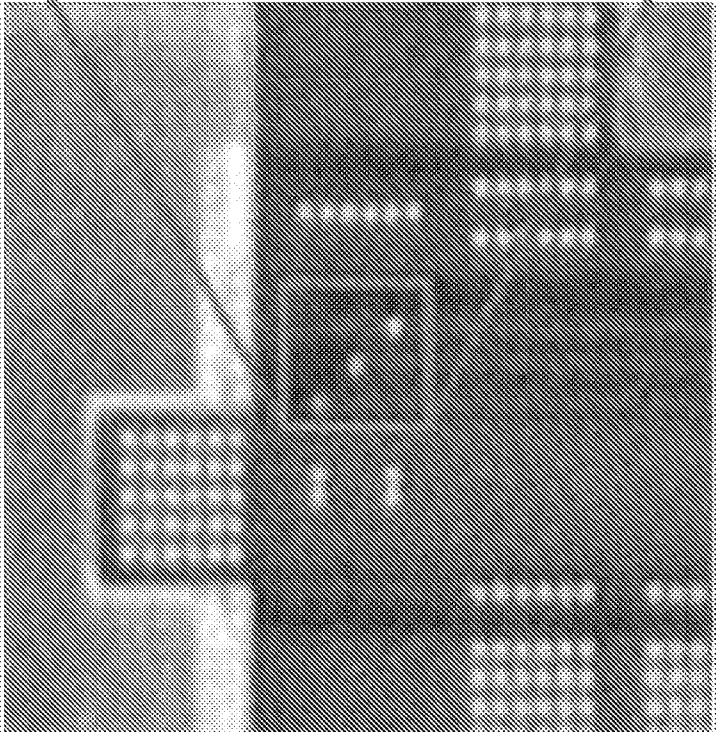


FIG. 3A

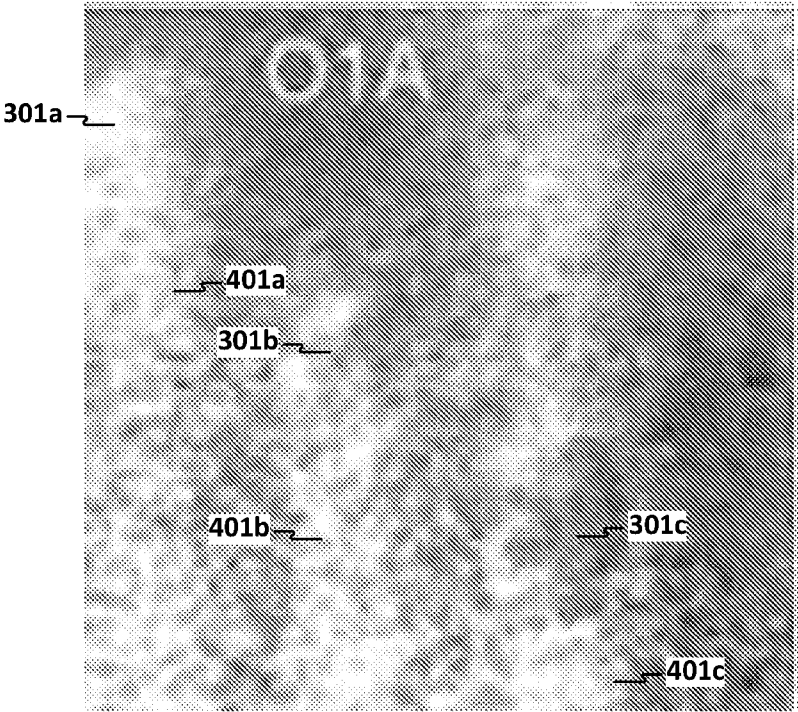


FIG. 4A

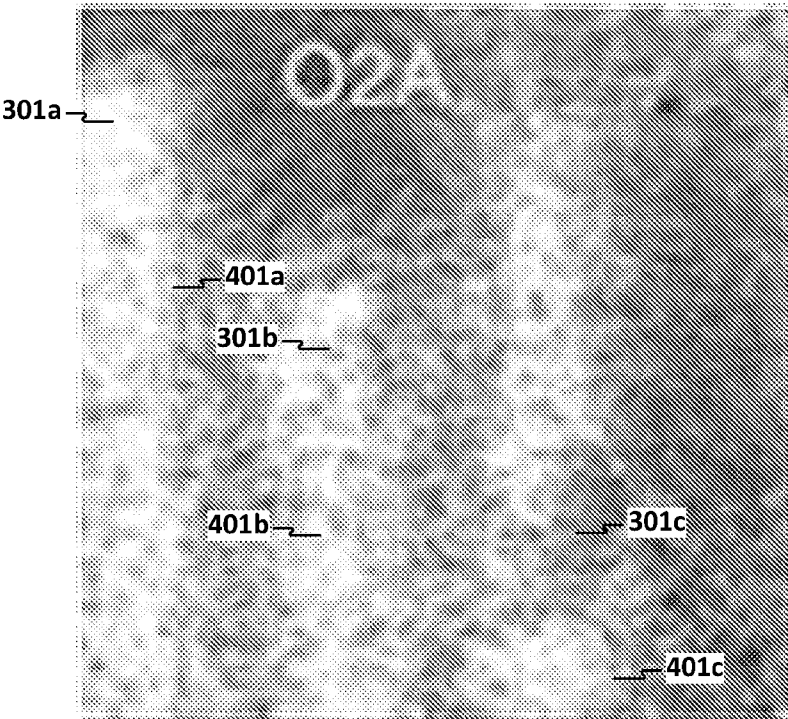


FIG. 4B

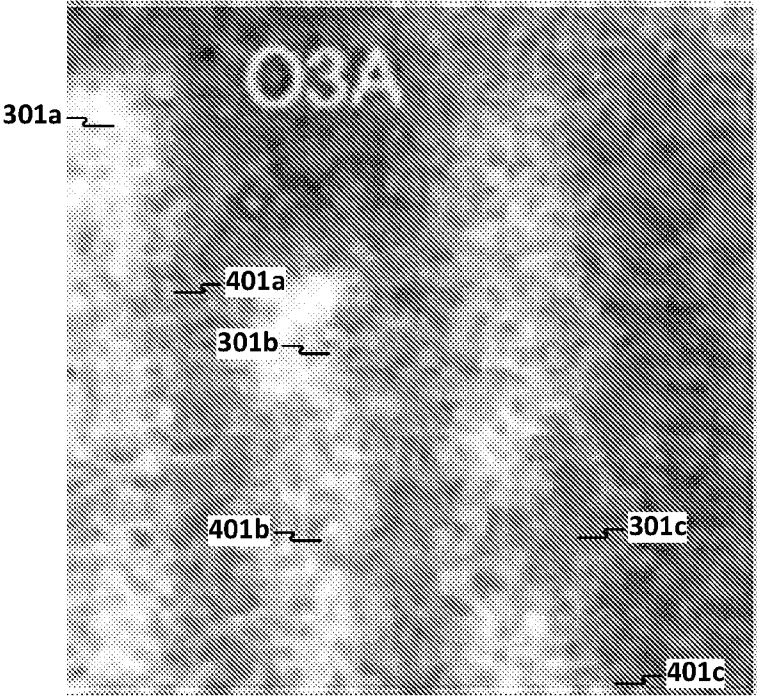


FIG. 4C

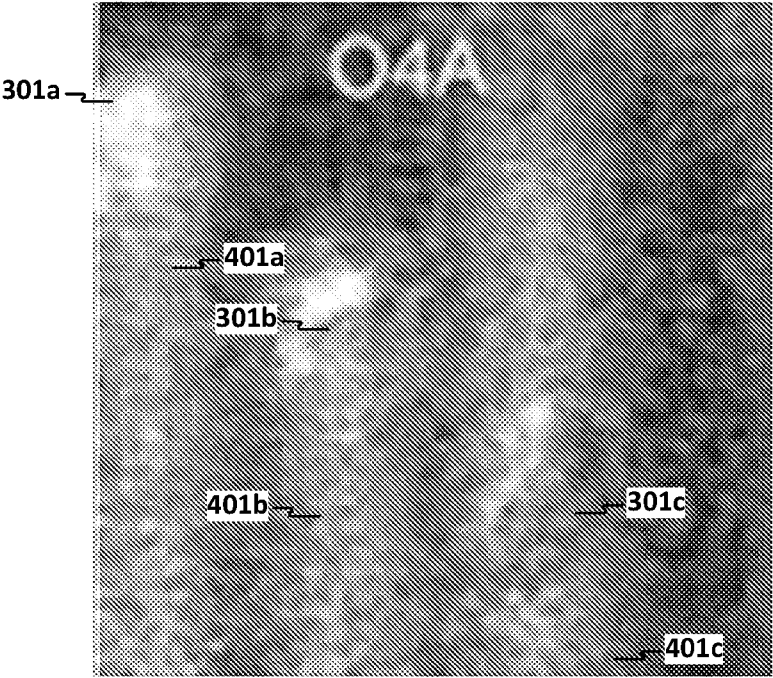


FIG. 4D

INTERNATIONAL SEARCH REPORT

International application No.

PCT/US 24/10366

A. CLASSIFICATION OF SUBJECT MATTER

IPC - INV. G01Q 60/06, G01Q 60/18, G01Q 60/34 (2024.01)

ADD. G01N 21/3581 (2024.01)

CPC - INV. G01Q 60/06, G01Q 60/18, G01Q 60/34

ADD. G01N 21/3581

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

See Search History document

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

See Search History document

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

See Search History document

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	CN 105628641 A (CHONGQING INSTITUTE OF GREEN AND INTELLIGENT TECHNOLOGY OF CAS) 01 June 2016 (01.06.2016) English machine translation, abstract, pages 2-4	1-16
Y	US 6,532,806 B1 (XIANG et al.) 18 March 2003 (18.03.2003) Fig 2, 2A, 3, 19, abstract, col 11, ln 26-31, col 24, ln 7-10	1-16
Y	US 2003/0211336 A1 (TSAI et al.) 13 November 2003 (13.11.2003) Fig 1-4, abstract, para [0015]	3, 6, 8, 10, 12, 15, 16

☐ 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

"D" document cited by the applicant in the international application

"E" earlier application or patent 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 citation or other special reason (as specified)

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

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

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

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

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

"&" document member of the same patent family

Date of the actual completion of the international search

29 March 2024 (29.03.2024)

Date of mailing of the international search report

APR 24 2024

Name and mailing address of the ISA/US

Mail Stop PCT, Attn: ISA/US, Commissioner for Patents
P.O. Box 1450, Alexandria, Virginia 22313-1450

Facsimile No. 571-273-8300

Authorized officer

Kari Rodriguez

Telephone No. PCT Helpdesk: 571-272-4300