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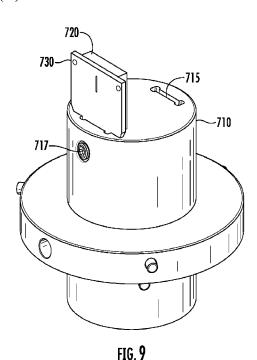
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(54) Title: APPARATUS AND METHOD FOR ION BEAM CROSS SECTION POLISHING OF EXTENDED SAMPLE REGIONS



(57) Abstract: A system and method for ion beam milling of a sample wherein the sample and a beam shield are rotated at a distance from a rotation axis of a sample stage during the ion beam milling.

APPARATUS AND METHOD FOR ION BEAM CROSS SECTION POLISHING OF EXTENDED SAMPLE REGIONS

CROSS REFERENCE TO RELATED APPLICATIONS

[0001] This application claims priority to and incorporates by reference in the entirety of United States Provisional Application Number 63/444,461, filed on February 9, 2023.

BACKGROUND OF THE INVENTION

[0002] The present disclosure relates to the use of one or more ion beams to prepare materials for microscopic observation or spectroscopic analysis. Microscopic observational techniques include, but are not limited to, optical microscopy, scanning electron microscopy (SEM), transmission electron microscopy (TEM), scanning transmission electron microscopy (STEM), reflection electron microscopy (REM). Spectroscopic analysis techniques include, but are not limited to, x-ray micro-analysis, reflection electron energy-loss spectroscopy (REELS), electron back-scattered diffraction (EBSD), x-ray photoelectron spectroscopy (XPS), and Auger electron spectroscopy (AES). Materials to be viewed under any microscopic technique may require processing to produce a sample suitable for microscopic examination. [0003] Ion beam milling of a material can produce samples that are well suited for microscopic examination. An ion beam irradiating device may generate, accelerate, and direct a beam of ions toward a sample. The impact of ions on the sample sputters material away from the area of ion impact. Furthermore, the sample surface may be polished by the ion beam to a substantially smooth condition further enhancing observational properties of the sample. Regions of interest in the sample may be exposed and polished by the use of ion beams thus making a suitable observational sample from the material under investigation. [0004] Broad Ion Beam Slope-Cutting (BIBSC), also known as cross section cutting using broad ion beam sources or cross section polishing using broad ion beam sources, is a rapid method for removing sample material to expose a smooth and substantially artifact-free cross-sectional surface for ultimate analysis by various microscopies and spectroscopies. A notable advantage of the BIBSC technique is high rates of surface preparation that can exceed tens or hundreds or thousands of square microns per hour, often over sample milling times of tens or hundreds of minutes.

[0005] Important considerations to users of the BIBSC technique include: reducing or minimizing the effort and time that the user is occupied in processing the sample; reducing or minimizing the number of steps where delicate samples are directly handled and at risk for damage, such as during mounting to sample holders for processing or analysis; reducing or minimizing the time and effort the user is occupied transferring the sample into the ultimate analysis equipment (imaging or spectroscopy), and aligning the coordinates of the prepared sample region to the ultimate analysis equipment prior to analysis; ensuring high quality and high probability of success in processing and imaging the sample; reducing or minimizing

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the time that the BIBSC ion milling equipment and sample mounting equipment are occupied for each sample; and ensuring high-quality microscopy observation of the sample during sample mounting and ultimate analysis by reducing the working distance required between the sample and the objective or probeforming lens used for observation.

BRIEF DESCRIPTION OF THE DRAWINGS

- [0006] FIG. 1 is a cross section diagram of an ion beam sample preparation apparatus;
- [0007] FIG. 2 is an isometric drawing of an ion beam shield and a sample prior to milling;
- [0008] FIG. 3 is an isometric drawing of an ion beam shield and a sample after milling;
- [0009] FIG. 4 is an isometric drawing of a sample and a sample shield mounted on a rotating stage for ion beam milling;
- [0010] FIG.5 is a side view cross-section view drawing of a sample being milled by ion beam milling;
- [0011] FIG. 6 is a top view drawing of a sample mounted at a distance from the rotation axis of a sample stage;
- [0012] FIG. 7 is a drawing of a sample mounted at a distance from the rotation axis of a sample stage;
- [0013] FIG. 8 is a drawing of a two-position sample stage;
- [0014] FIG. 9 is a further drawing of a two-position sample stage;
- [0015] FIG. 10 is a top view of a milled sample and a sample shield;
- [0016] FIG. 11 is drawing of a three-position sample stage with an additional center position;
- [0017] FIGS. 12A-12F are diagrams of a milling process for a two-position sample stage;
- [0018] FIGS. 12G-12H are diagrams of a milling process for a further two-position sample stage;
- [0019] FIGS. 12I-12J are diagrams of a milling process for a four-position sample stage; and
- [0020] FIG. 13 is a diagram of a controller device.

DETAILED DESCRIPTION

[0021] It will be understood that, although the terms first, second, third, etc. may be used herein to describe various elements, these elements should not be limited by these terms. These terms may be used to distinguish one element from another. For example, a first element may be termed a second element and a second element may be termed a first element without departing from the scope of the present invention. As used herein, the term "and/or" may include any and all combinations of one or more of the associated listed items.

[0022] It will be understood that when an element such as a layer, region, or substrate is referred to as being "on" or extending "onto" another element, it may be directly on or extend directly onto the other element or intervening elements may also be present. In contrast, when an element is referred to as being "directly on" or extending "directly onto" another element, there may be no intervening elements present. It will also be understood that when an element is referred to as being "connected" or "coupled" to another

element, it may be directly connected or coupled to the other element and/or connected or coupled to the other element via one or more intervening elements. In contrast, when an element is referred to as being "directly connected" or "directly coupled" to another element, there are no intervening elements present between the element and the other element. It will be understood that these terms are intended to encompass different orientations of the element in addition to any orientation depicted in the figures.

[0023] Relative terms such as "below," "above," "upper,", "lower," "horizontal" or "vertical" may be used herein to describe a relationship of one element, layer, or region to another element, layer, or region as illustrated in the figures. It will be understood that these terms are intended to encompass different orientations of the device in addition to the orientation depicted in the figures.

[0024] With reference to Figure 1, there is shown a schematic cross section view of an embodiment of an ion beam sample preparation apparatus 2. The apparatus includes: a vacuum chamber 10 in which a sample 8 is prepared; chamber cover 18, which seals vacuum chamber 10 from the outside atmosphere; vacuum pump means 90 and pumping manifold 92, which together bring vacuum chamber 10 to vacuum levels appropriate for ion beam milling; tilting ion beam irradiating means 36 and tilt drive 38, which creates and directs an ion beam having a central ion beam axis 22 toward sample 8; a shield 60, which shields at least a portion of sample 8 from at least a portion of the ion beam; a shield retention stage 40, which holds and accurately positions shield 60 with respect to the direction and extent of the ion beam; a shield retention means 42, which both retains shield 60 in shield retention stage 40, and also urges shield 60 to remain in a position whereby the ion beam may prepare sample 8.

[0025] The ion beam 22 preferably comprises noble gas ions. Elements used for the ion beam may include but are not limited to: Argon, Xenon, and Krypton. The ion beam may also comprise a mixture of ions and neutrals. Shield retention stage 40 is disposed in vacuum chamber 10 in a predetermined position and orientation with respect to central ion beam axis 22. Tilting ion beam irradiating means 36 and tilt drive 38 enable the ion beam to prepare the sample by directing central ion beam axis 22 toward sample 8 from more than one tilt angle with respect to shield 60. FIG. 1 shows tilting ion beam irradiating means 36 operating at a first tilt angle.

[0026] FIG. 2 is a perspective view of shield 60, shielding surface 61, sample 8 durably adhered to the shield, and visible alignment mark 65. FIG. 11A depicts the sample prior to ion beam preparation. FIG. 3 is a perspective view of the same objects depicted in FIG. 2. However, FIG. 3 represents the sample after ion beam sample preparation. Shielding surface 61 intercepts a portion of the ion beam, which travels along central ion beam axis 22. A portion of sample 8 is sputtered away by the ion beam during sample preparation, thereby exposing a portion of the sample lying in the plane defined by shield edge 63 and central ion beam axis 22. A sample prepared in this way will be suitable for observation or analysis with a variety of microscopic or spectroscopic techniques, particularly those requiring a highly polished planar

surface. A milling tool as described above removes material from a sample over a region based on the size of the ion beam, typically between 0.5 and 1.0 mm in width at the sample surface.

[0027] FIG. 4 shows a perspective schematic view of rotating shield retention stage 50, on which sample 8 has been durably adhered to shield 60 prior to placing the shield and sample combination in a shield retaining position of rotating shield retention stage 50. Shield 60 has a shielding surface 61, which is positioned in relation to sample 8 to shield at least a portion of said sample 8 from at least a portion of the ion beam. Rotation drive 52 enables rotating shield retention stage 50 to rotate about rotation axis 54.

[0028] FIG. 5 is a side view showing the portion of a sample 510 that is milled by the ion beam 520 past the edge of the shielding blade 530. Note that in this view the axis of rotation of the sample stage is about an axis that lies between the sample and the shielding blade.

[0029] FIG. 6 is a top view of an ion beam milling stage 610 according to an embodiment. In this embodiment, the sample 620 and blade 630 interface is placed off of the rotation axis 612 so that the sample 620 and blade 630 are swept in front of the ion beam 640 as the stage 610 rotates, thereby creating a wider cut on the sample 620.

[0030] In embodiments, the ion beam 640 is turned on when the shield blade 630 is between the ion gun (not shown) and the rotation axis 612, and off when it is not. Thus, a sample cross section can be created that is the total width of the shield blade 630. In embodiments, when a narrower cut than the width of the shield blade is desired, the ion beam can be turned off during a smaller set of rotation angles such that any width can be created between the minimum width of the beam and the full width of the blade (14 mm). In embodiments, a user interface may be defined so that a user enters a cut width and the software translates that into a set of angles where the ion beam is turned on.

[0031] In embodiments, the sample is milled in one pass with the stage 610 rotated in a single direction so that the sample 620 is cut beginning at one end and ending at the opposite end of the sample. In further embodiments, the stage may be rotated back and forth one or more times so that the sample is first cut from right to left, for example, then the stage direction is reversed and the sample is cut from left to right. In embodiments, the ion beam may be turned off at the end of each pass and turned back on when the stage rotates in the opposite direction.

[0032] In embodiments, the distance from the sample/shield interface to the rotation axis of the sample stage is 7 mm. This distance allows for cutting the full width of a standard shield blade (~14 mm) and allows an optical microscope to image the sample through the standard load lock window. In further embodiments, other distances between the sample/shield interface and the rotation axis can be used. Smaller distances result in the beam striking the sample at larger angles from normal incidence when the beam is striking away from the center of the sample. Larger distances place the sample closer to the ion beam gun(s) so the current density and milling rate is larger, e.g. for a divergent beam.

[0033] FIG. 7 is a three-dimensional view of an ion beam milling stage 710 with a sample 720 and a shield blade 730 mounted at a distance from the rotation axis 712, according to an embodiment.

[0034] FIGS. 8 and 9, show embodiments wherein the shield blade 730 is mounted to the sample stage 710 by insertion into a slot 715 and fastened by a set screw 717 in the sample stage that is tightened from the side of the sample stage. In embodiments, the shield blade is generally "T" shaped having shoulders 731, 732 that sit flush against the sample stage when the shield blade is fully inserted. In embodiments, the plane of the shield blade is parallel to the rotation axis of the sample stage. In further embodiments the plane of the shield blade is within +/- 10 degrees of parallel to the rotation axis of the sample stage. In embodiments, the sample stage may have more than one slot 715 for mounting a shield blade. As shown in FIGS. 8 and 9, two slots 715 are arranged opposite each other.

[0035] FIG. 10 is a top view of a sample 1020 attached to a shield 1030. In this view, the sample 1020 has been milled by an ion beam as described above with respect to FIG.s 6 and 7. Because the sample 1020 and shield 1030 were mounted at a distance from the center of rotation of a sample stage, the milled portion 1022 of the sample extends along most of the width of the sample 1020. Depending on the relative widths of the sample and the shield blade, the entire width of the sample may be milled in this matter if desired. This may be contrasted with FIG. 3, wherein the sample 8 and shield blade 61 are rotated about the center of rotation of the sample stage. In FIG. 3, a much more limited width of the sample is shown as being milled.

[0036] FIG. 11 shows an embodiment wherein the sample stage 1110 is configured to hold three shield blades 1120, 1122 and 1124. Each of the shield blades is shown with a respective sample for milling affixed 1121, 1123, and 1125. Holes 1114, 1112 for set screws for fastening shield blades 1120 and 1124, respectively, are shown in this view.

[0037] FIGS. 12A-12F show an exemplary sequence for milling two samples 1212 and 1211 mounted on the same stage in a system having first 1221 and second 1222 ion beam guns. A fully black arrow indicates an ion beam gun that is turned on and an arrow with a white interior indicated an ion beam gun that is turned off. In FIG. 12A, the first sample is being milled by the right-side ion beam gun 1222. The left-side gun 1221 is off. As the stage is rotated (counterclockwise) to the position in FIG. 12B, the right-side gun stays one and continues milling the first sample 1211. The left side gun is off. As the stage is further rotated to the position shown in FIG. 12C, both guns 1221, 1222 are turned off as neither is aimed at a sample in this position. As the stage is further rotated counterclockwise to the position shown in FIG. 12D, the left-side gun 1221 is turned on and is milling the second sample 1212. The right-side gun remains off. As the stage is further rotated to the position shown in FIG. 12E both guns 1221, 1222 are again turned off as neither is aimed at a sample. Lastly, in FIG. 12F, the left-side gun 1221 is turned on and milling the first sample 1211 again and the right-side gun 1222 is turned off.

[0038] FIGS. 12G and 12H show an exemplary sequence for milling two samples 1231 and 1232 mounted on the same stage at 90-degree angles to each other in a system having first 1221 and second 1222 ion beam guns. While this embodiment shows the samples mounted at 90 degrees to each other, different angular spacings are possible and within the scope of the disclosure. In FIG. 12G, both samples are exposed to ion beams as both ion beam guns 1221, 1222 are turned on. Sample 1231 is exposed to ion beam gun 1221 and sample 1232 is exposed to ion beam gun 1222. In FIG. 12H, the sample stage is rotated counter-clockwise with respect to FIG. 12G and at this stage in the process, both ion beam guns 1221 and 1222 are turned off as neither sample is facing either ion beam gun. In embodiments, the arrangement of FIGS. 12G and 12H may provide an overall faster milling rate for the two samples than other embodiments shown herein.

[0039] FIGS. 12I and 12J show an exemplary sequence for milling four samples 1241, 1242, 1243 and 1244 mounted to a single sample stage. Similar to the embodiment shown in FIGS. 12G and 12H, the samples are mounted at 90 degrees to each other. While this embodiment shows the samples mounted at 90 degrees to each other, different angular spacings are possible and within the scope of the disclosure. Thus, it is possible to mill two samples at a time (the two closest to the ion beam guns, e.g. 1242, 1243) with two ion beam guns 1221, 1222 turned on at the same time as shown in FIG. 12I. In FIG. 12J, the sample stage is rotated counter-clockwise to a position where no samples are facing a ion beam gun and both guns 1221, 1222 are turned off.

[0040] FIG. 13 is a diagram illustrating exemplary physical components of a device 1300. Device 1300 may correspond to various devices within the above-described system, such as a system controller for controlling, for example, the ion beams and the rotation stage. Device 1300 may include a bus 1310, a processor 1320, a memory 1330, an input component 1340, an output component 1350, and a communication interface 1360.

[0041] Bus 1310 may include a path that permits communication among the components of device 1300. Processor 1320 may include a processor, a microprocessor, or processing logic that may interpret and execute instructions. Memory 1330 may include any type of dynamic storage device that may store information and instructions, for execution by processor 1320, and/or any type of non-volatile storage device that may store information for use by processor 1320.

[0042] Software 1335 includes an application or a program that provides a function and/or a process. Software 1335 is also intended to include firmware, middleware, microcode, hardware description language (HDL), and/or other form of instruction. By way of example, with respect to the network elements that include logic to provide proof of work authentication, these network elements may be implemented to include software 1335.

[0043] Input component 1340 may include a mechanism that permits a user to input information to device 1300, such as a keyboard, a keypad, a button, a switch, etc. Output component 1350 may include a mechanism that outputs information to the user, such as a display, a speaker, one or more light emitting diodes (LEDs), etc.

[0044] Communication interface 1360 may include a transceiver that enables device 1000 to communicate with other devices and/or systems via wireless communications, wired communications, or a combination of wireless and wired communications. For example, communication interface 1360 may include mechanisms for communicating with another device or system via a network. Communication interface 1360 may include an antenna assembly for transmission and/or reception of RF signals. In one implementation, for example, communication interface 1360 may communicate with a network and/or devices connected to a network. Alternatively, or additionally, communication interface 1360 may be a logical component that includes input and output ports, input and output systems, and/or other input and output components that facilitate the transmission of data to other devices.

[0045] Device 1300 may perform certain operations in response to processor 1320 executing software instructions (e.g., software 1335) contained in a computer-readable medium, such as memory 1330. A computer-readable medium may be defined as a non-transitory memory device. A non-transitory memory device may include memory space within a single physical memory device or spread across multiple physical memory devices. The software instructions may be read into memory 1330 from another computer-readable medium or from another device. The software instructions contained in memory 130 may cause processor 1320 to perform processes described herein, for example, the ion beam and stage rotation sequence described with respect to FIGS. 12A-12F. Alternatively, hardwired circuitry may be used in place of or in combination with software instructions to implement processes described herein. Thus, implementations described herein are not limited to any specific combination of hardware circuitry and software.

[0046] Device 1300 may include fewer components, additional components, different components, and/or differently arranged components than those illustrated in FIG. 13. As an example, in some implementations, a display may not be included in device 1300. In these situations, device 1300 may be a "headless" device that does not include input component 1340. Additionally, or alternatively, one or more components of device 1300 may perform one or more tasks described as being performed by one or more other components of device 1300.

[0047] Having described the embodiments in detail, those skilled in the art will appreciate that, given the present description, modifications may be made to the embodiments described herein without departing from the spirit of the inventive concept. Therefore, it is not intended that the scope of the invention be limited to the specific embodiments illustrated and described.

CLAIMS

1. A method of preparing a sample having a first planar surface by ion beam milling comprising:

aligning the sample with the first planar surface adjacent to an ion beam shield such that a portion of the first planar surface is exposed beyond the ion beam shield,

mounting the sample and ion beam shield onto a rotating sample stage the first planar surface being located at a first non-zero distance from the axis of rotation and the ion beam shield being located farther from the axis of rotation than the sample;

irradiating a first area on the portion of the first planar surface that is exposed beyond the ion beam shield with an ion beam; and

rotating the rotating sample stage by a first rotation angle so as to irradiate a second area of the portion of the first planar surface that is exposed beyond the ion beam shield with the ion beam.

- 2. The method of claim 1, wherein the first planar surface is mounted parallel to an axis of rotation of the rotating sample stage within plus or minus 10 degrees.
- 3. The method of claim 1, wherein the first non-zero distance is one half of a width of the ion beam shield.
 - 4. The method of claim 3, wherein the ion beam shield is 14 mm wide.
- 5. The method of claim 1, wherein the rotating sample stage is rotated to a plurality of rotation angles and the sample is irradiated by the ion beam for each of the plurality of rotation angles.
- 6. The method of claim 5, where in the plurality of rotation angles are between a first rotation angle limit and a second rotation angle limit.
- 7. The method of claim 6, further comprising: determining the first rotation angle limit and the second rotation angle limit based on the first non-zero distance, a width of the ion beam shield and a desired milling width of the sample.
- 8. The method of claim 6, further comprising rotating the sample stage back and forth between the first and second rotation angles a plurality of times.
 - 9. An apparatus for preparing a sample by ion beam milling comprising:
 - a first ion beam emitter;
 - a rotating sample stage; and
 - a sample shield:

the rotating sample stage comprising:

- a rotation axis;
- a sample mounting surface;

a first sample shield mounting slot located on the sample mounting surface at a first non-zero distance from the rotation axis;

the sample shield having a first planar surface configured for mounting a sample thereon and having a first portion configured for insertion in the sample shield mounting slot, the sample shield mounting slot being configured such that the first planar surface is parallel to the rotation axis within plus or minus 10 degrees;

the sample shield configured to shield a first portion of the sample from the ion beam and to expose a second portion of the sample to the ion beam;

the rotating sample stage configured to expose a first sub-portion of the sample to the ion beam with the rotating sample stage at a first rotation angle and to expose a second sub-portion of the sample to the ion beam with the rotating sample stage at a second rotation angle.

10. The apparatus of claim 9 wherein the rotating sample stage is configured to rotate between a first rotation angle limit and a second rotation angle limit,

the first rotation angle limit and the second rotation angle limit being based on the first non-zero distance, a width of the sample shield and a desired milling width of the sample.

11. The apparatus of claim 9 wherein the rotating sample stage comprises a second shield mounting slot located on the sample mounting surface,

the second shield mounting slot configured to place a second sample shield a second non-zero distance from the rotation axis and the second shield mounting slot being configured such that a second planar surface of the second sample shield is parallel to the rotation axis within plus or minus 10 degrees.

- 12. The apparatus of claim 11, wherein the first non-zero distance and the second non-zero distance are the same.
- 13. The apparatus of claim 11 wherein the rotating sample stage comprises a third shield mounting slot located on the sample mounting surface,

the third mounting slot configured to place a third sample shield a third non-zero distance from the rotation axis and the third shield mounting slot being configured such that a third planar surface of the third sample shield is parallel to the rotation axis within plus or minus 10 degrees.

- 14. The apparatus of claim 13, wherein the first non-zero distance, the second non-zero distance and the third non-zero distance are the same.
- 15. The apparatus of claim 13 wherein the rotating sample stage comprises a fourth shield mounting slot located on the sample mounting surface,

the fourth mounting slot configured to place a fourth sample shield a third non-zero distance from the rotation axis and the fourth shield mounting slot being configured such that a fourth planar surface of the fourth sample shield is parallel to the rotation axis within plus or minus 10 degrees.

16. The apparatus of claim 15, wherein the first non-zero distance, the second non-zero distance the third non-zero distance and the fourth non-zero distance are the same.

- 17. The apparatus of claim 9, further comprising a second ion beam gun and wherein said first and second ion beam guns are configured to be turned on only when directed at a sample.
- 18. The apparatus of claim 11, further comprising a second ion beam gun and wherein said first and second ion beam guns are configured to be turned on only when directed at a sample.
- 19. The apparatus of claim 13, further comprising a second ion beam gun and wherein said first and second ion beam guns are configured to be turned on only when directed at a sample.
- 20. The apparatus of claim 15, further comprising a second ion beam gun and wherein said first and second ion beam guns are configured to be turned on only when directed at a sample.

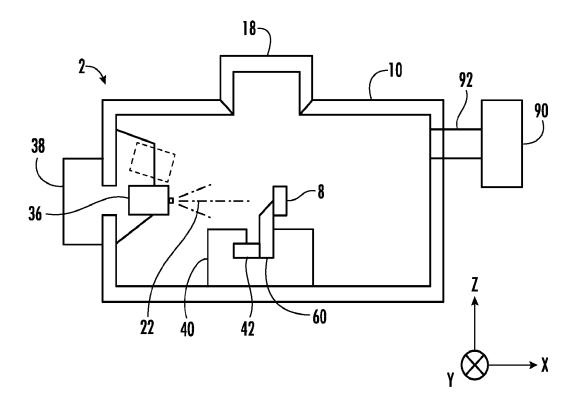
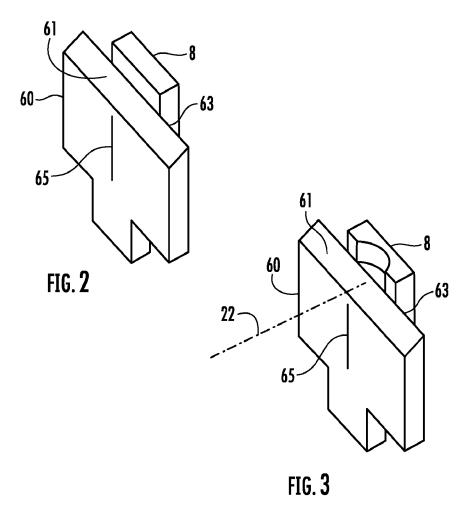


FIG. 1



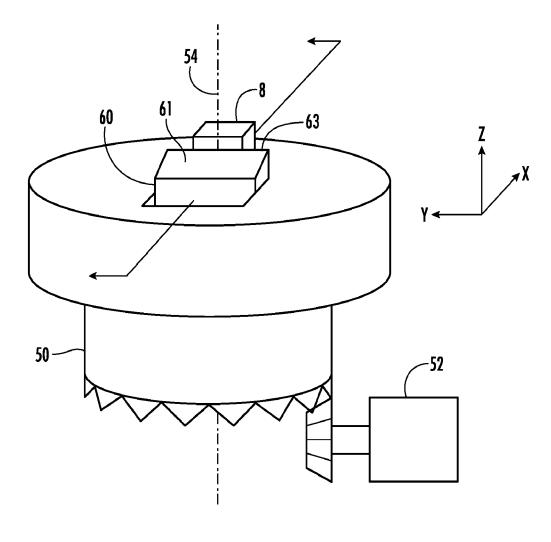


FIG. 4

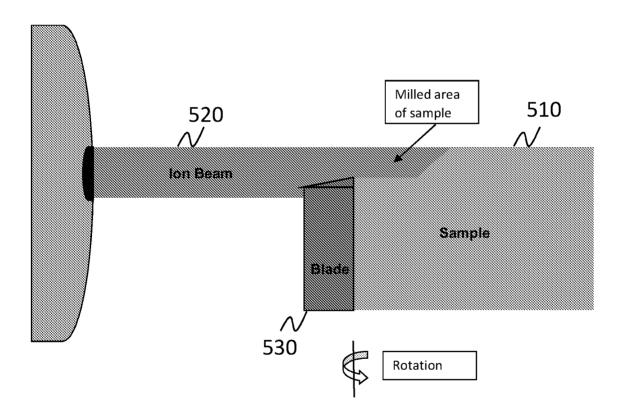


FIG. 5

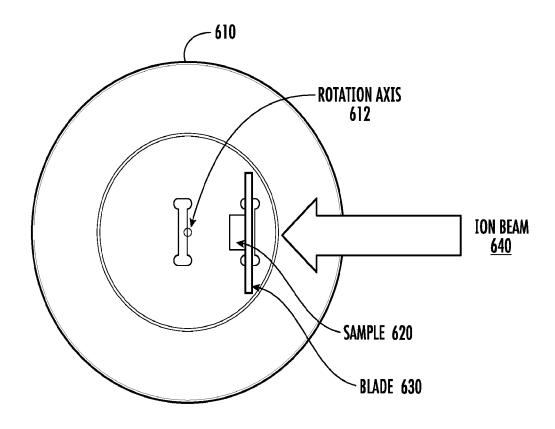
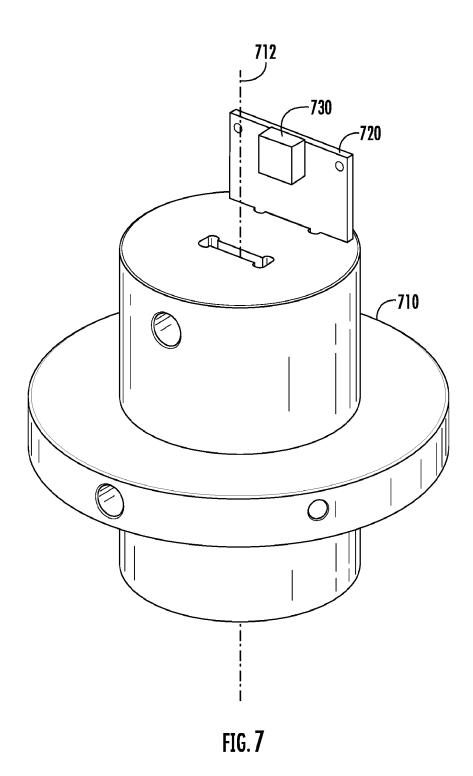
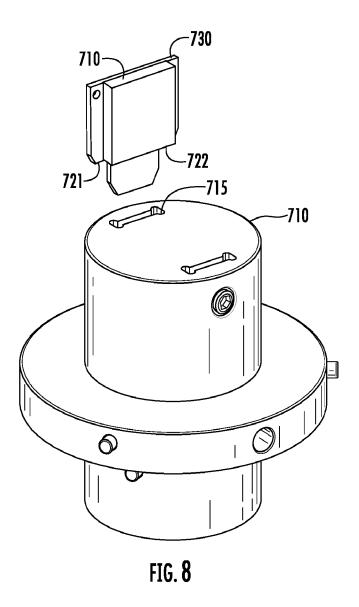
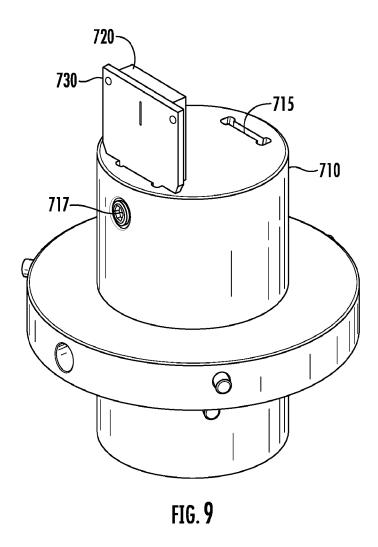


FIG. **6**







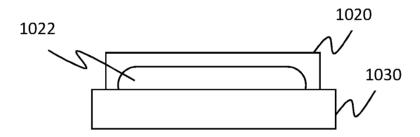


FIG. 10

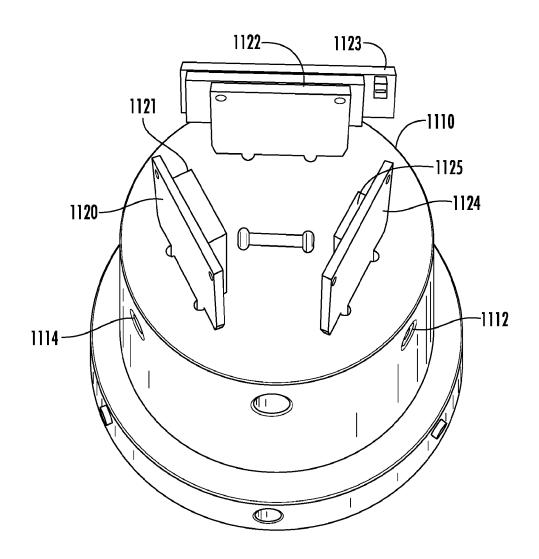
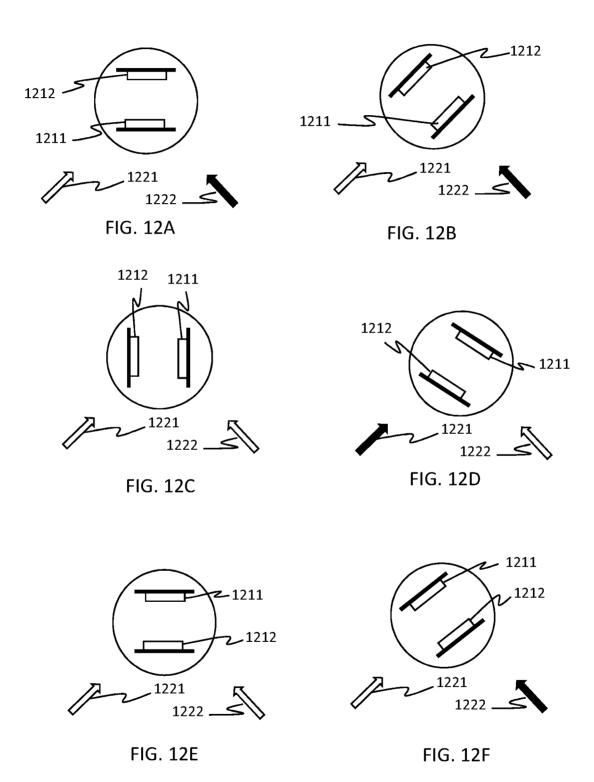
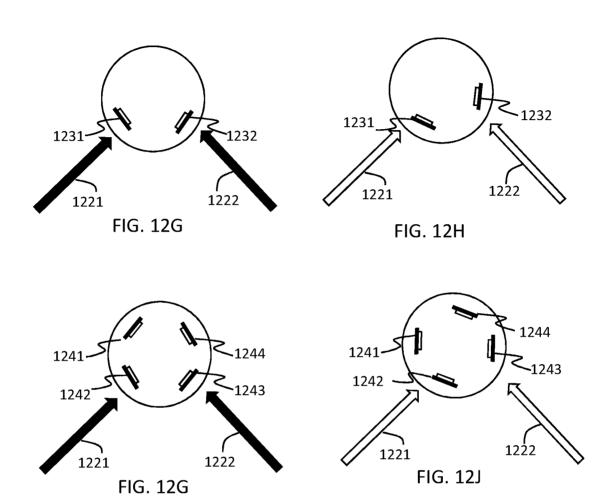
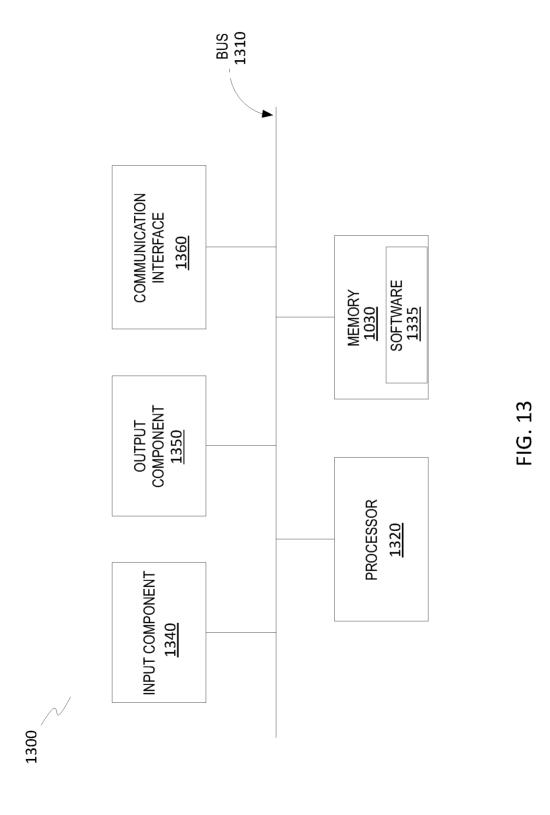


FIG. 11







INTERNATIONAL SEARCH REPORT

International application No. PCT/US24/14817

| A. CLASSIFICATION OF SUBJECT MATTER | | | |
|---|---|---|-------------------------------|
| | INV. G01N 1/44; B23K 15/08; G01N 1/32; H01J 37/31 (2023.01) | | |
| | ADD. | (2023.01) | |
| | INV. G01N 1/44; B23K 15/08; G01N 1/32; H01J 37/31 | | • |
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| ADD. | | | |
| 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 database consulted during the international search (name of database and, where practicable, search terms used) See Search History document | | | |
| C. DOCUMENTS CONSIDERED TO BE RELEVANT | | | |
| Category* | Citation of document, with indication, where a | appropriate, of the relevant passages | Relevant to claim No. |
| X | US 2022/0277925 A1 (JEOL, LTD.) 01 September 2022; figures 1-9, 11, 22-25; paragraphs [0062, 0064, 0067, 0069-0074, 0092-0093, 0096-0102, 0110-0114, 0117-0120, 0130, | | 1-16 |
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| Furthe | r documents are listed in the continuation of Box C. | See patent family annex. | |
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