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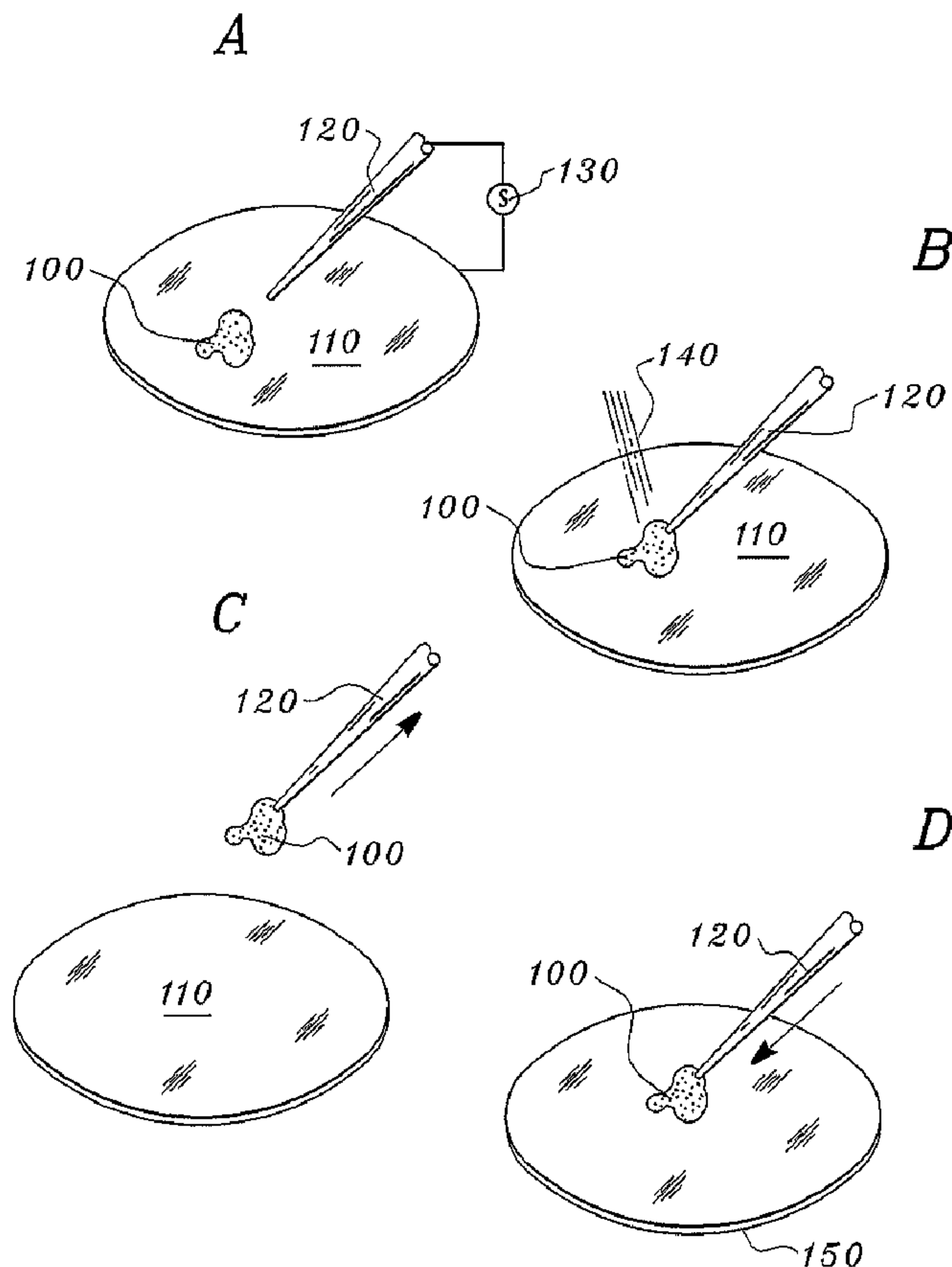
(54) Titre : PROCEDE DE MANIPULATION DE PARTICULES MICROSCOPIQUES ET D'ANALYSE DE LA COMPOSITION DE CELLES-CI
(54) Title: METHOD FOR MANIPULATING MICROSCOPIC PARTICLES AND ANALYZING THE COMPOSITION THEREOF




(57) Abrégé/Abstract:
We disclose a method for analyzing the composition of a microscopic particle (100) resting on a first sample surface (110). The method comprises positioning a micro-manipulator probe (120) near the particle (100); attaching the particle (100) to the probe

(57) **Abrégé(suite)/Abstract(continued)**:
(120); moving the probe (120) and the attached particle (100) away from the first sample surface (110); positioning the particle (100) on a second sample surface (150); and, analyzing the composition of the particle (100) on the second sample surface (150) by energy-dispersive X-ray analysis or detection of Auger electrons. The second surface (150) has a reduced or non-interfering background signal during analysis relative to the background signal of the first surface (110). We also disclose methods for adjusting the electrostatic forces and DC potentials between the probe (120), the particle (100), and the sample surfaces (110, 150) to effect removal of the particle (100), and its transfer and relocation to the second sample surface (150).

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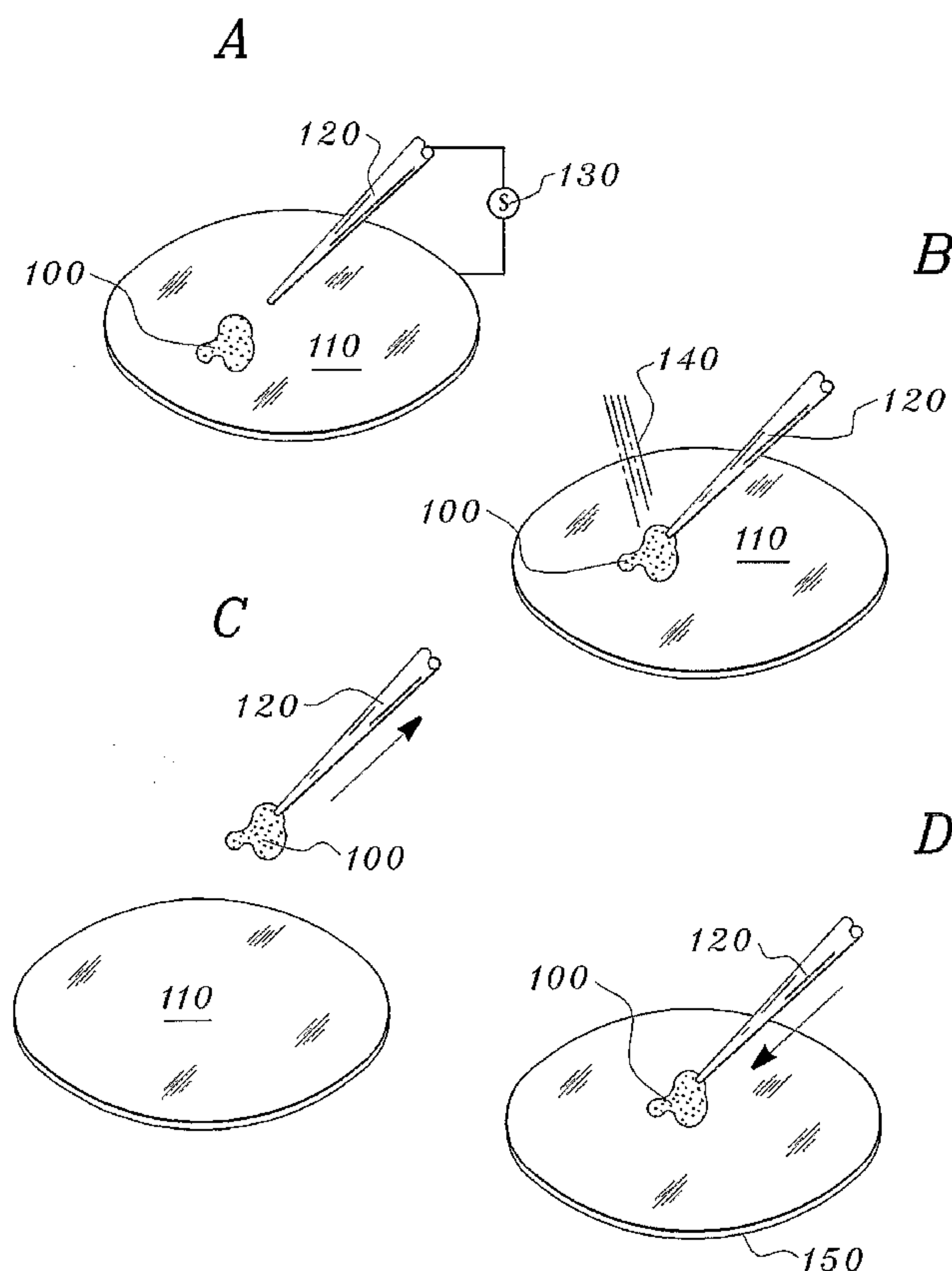
# (54) Title: METHOD FOR MANIPULATING MICROSCOPIC PARTICLES AND ANALYZING THE COMPOSITION THEREOF




**(57) Abstract:** We disclose a method for analyzing the composition of a microscopic particle (100) resting on a first sample surface (110). The method comprises positioning a micro-manipulator probe (120) near the particle (100); attaching the particle (100) to the probe (120); moving the probe (120) and the attached particle (100) away from the first sample surface (110); positioning the particle (100) on a second sample surface (150); and, analyzing the composition of the particle (100) on the second sample surface (150) by energy-dispersive X-ray analysis or detection of Auger electrons. The second surface (150) has a reduced or non-interfering background signal during analysis relative to the background signal of the first surface (110). We also disclose methods for adjusting the electrostatic forces and DC potentials between the probe (120), the particle (100), and the sample surfaces (110, 150) to effect removal of the particle (100), and its transfer and relocation to the second sample surface (150).

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10 **METHOD FOR MANIPULATING MICROSCOPIC PARTICLES AND**
11 **ANALYZING**
12 **THE COMPOSITION THEREOF**
13
14 **Patent Application of**
15 **Thomas M. Moore and John M. Anthony**
16
17 **TECHNICAL FIELD**
18 The invention relates to techniques for removing and analyzing microscopic
19 particles from a sample surface, particularly from semiconductor samples.
20 **BACKGROUND**
21 In the semiconductor industry, unexpected particles due to contamination will
22 cause yield loss during the manufacturing process. Since a major focus of this industry is
23 aggressive reduction in feature size for pattern line widths, the minimum size of particles
24 that can cause performance loss also decreases rapidly. A reasonable estimate for a
25 "killer defect" size is that greater than one-third the size of the smallest feature on the
26 semiconductor wafer.
27 Although semiconductor manufacturing is performed in clean rooms with
28 stringent particle standards, unexpected contamination will occur due to sources such as
29 moving parts, human presence, gas condensation, and chamber wear. Control and
30 removal of these particles is a continuous process. In many cases, removal of the source
31 of particles requires an understanding of their origin. Many of these particles or defects
32 are too small for detection in a general purpose optical inspection microscope, so higher
33 resolution methods are required, using charged-particle microscopes, such as scanning

1 electron microscopes (SEM), transmission electron microscopes (TEM), scanning Auger
2 microprobes (SAM), or focused ion beam (FIB) instruments, are required.

3 Even an image of the particle is usually insufficient to trace the origin of the
4 particle, and more information is required. Elemental composition is valuable in
5 identifying the defect. This can be done in various ways using the charged-particle
6 systems mentioned above. Unfortunately, most of the analytical methods are limited by
7 background signals from the environment of the particle.

8 Throughput is also a critical parameter in semiconductor manufacturing.
9 Existing strategies for compositional analysis of particles on a semiconductor wafer, for
10 example, usually require removal of the wafer from the fabrication area for off-line
11 analysis using methods such as those described below. Removal from the line severely
12 reduces the throughput of the manufacturing process.

13 Particle identification on sample surfaces using electron-beam based identification
14 is complicated by the size of the particle relative to the electron penetration depth, and by
15 the nature of surrounding materials in the sample. As an electron beam interacts with
16 bulk solid materials, it expands to fill a teardrop-shaped volume as it loses energy. As
17 the primary beam interacts with atoms in this volume, it generates low energy Auger
18 electrons and X-rays that are characteristic of the elements involved.

19 The particular X-ray line generated will depend on the atomic number of the
20 element, the energy of the electron during the interaction, and other factors. When
21 trying to identify an unknown particle using conventional Energy-dispersive X-ray
22 Spectrophotometry (EDS), the energy of the electron beam must be large enough to
23 generate inner-shell X-rays from all possible relevant elements, which, for semiconductor
24 applications, may include elements of high atomic number such as tungsten.
25 Unfortunately, this energy results in a penetration depth that may be much larger than the
26 particle of interest, resulting in X-ray generation from the sample surface. These X-rays
27 interfere with any signal from the particle, making unique identification of the particle
28 material difficult. Conventional strategies for solving this problem involve either
29 resolving the X-ray lines of different elements, or reducing the energy of the exciting
30 electron beam.

31 For example, it is possible to detect and analyze electron-beam generated X-rays
32 from a particle by measuring the intensity and diffraction angle of the X-rays diffracted
33 by a reference crystal, or Wavelength Dispersive X-ray Spectrometry (WDS). One

1 chooses the crystal atomic spacing to deflect (with very high resolution) X-rays of a
2 given energy, thus allowing separation between X-ray lines of different elements. This
3 method has higher energy resolution than EDS but much slower throughput. In addition,
4 if the particle could be, as it often is, of the same composition as the sample surface, this
5 method will not uniquely determine the particle composition.

6 Other solutions involve reducing the energy of the primary electron beam to
7 guarantee the activated volume is less than the volume of the particle of interest. This
8 reduction in primary electron-beam energy results in characteristic X-rays of much lower
9 energy (M or L shell X-rays, rather than K shell). Conventional cooled semiconductor-
10 based detectors use the generation and collection of electron-hole pairs as a measure of
11 the energy of the ionizing radiation (a few eV for each electron-hole pair, depending on
12 the detecting material). A reduction in the X-ray energy therefore leads to a reduced
13 number of electron-hole pairs and reduced sensitivity to the particle material. In addition
14 the resolution of these detectors is governed by the statistics of the electron-hole
15 generation process, and reducing the energy of the detected X-ray often leads to
16 ambiguous identification of the element of interest. X-ray micro-calorimeter methods
17 have been used to detect these weak X-ray signals, using heat transferred to the detector
18 rather than the generation of electron-hole pairs. This process does allow measurement
19 of small X-ray energies, but micro-calorimeter instruments are expensive, have
20 complicated cooling requirements, and are slow compared to other methods. Also, the
21 electron beam must be kept smaller than the smallest dimension of the particle of interest,
22 rendering the method impractical for small, unsymmetrical particles.

23 Scanning Auger microprobe analysis also uses an electron beam to irradiate a
24 particle of interest, but rather than detecting any X-rays generated it focuses on the
25 detection of Auger electrons ejected from the atoms of the material. These Auger
26 electrons come from outer shells and have relatively low energies. The Auger electron
27 energies from a material produce a pattern that is characteristic of each element in the
28 material, and the shape and exact energy of the Auger transitions provide information on
29 the chemical bonding of the elements in the material (such as, phase or compound
30 information). The escape depth of these electrons is quite small (a few nm), so Auger
31 analysis focuses mainly on the surface of a sample. This is an advantage for the analysis
32 of small diameter particles (<10 nm). For the analysis of larger particles, one can
33 generate depth profiles by using an ion beam to sputter through the particle and take

1 periodic measurements, but this is inherently destructive of the surrounding sample due
2 to ion milling in the SAM, and requires background analyses on the sample near the
3 location of the particle. Auger analysis is typically more sensitive to light elements than
4 standard EDS analysis, making it more suitable to identify organic materials. However,
5 to improve counting statistics, high electron beam currents are typically employed. This
6 exaggerates the issues of thermo-mechanical drift and drift due to electrical charging of
7 the sample. This means that operating the SAM in the "spot mode," with the electron
8 beam positioned on the particle, involves a risk that over time the electron beam spot will
9 drift onto the sample that surrounds the particle. And the use of a raster pattern for the
10 electron beam will be more tolerant of drift for keeping the beam on the particle, but will
11 involve significant contamination of the results with signal from the surrounding material.
12 In either case, background contamination of the Auger results is a serious issue, and
13 Auger analyses of the surrounding material are required to uniquely identify the signal
14 from the particle. The acquisition of background analyses reduces throughput and
15 inherently damages the sample.

16 TEM can often be used for analysis of particles in or on surfaces. There are a
17 variety of methods for isolating the particle for analysis, including replication, lift-out or
18 cross sectioning the area of interest. These methods all destroy the sample surface and
19 must be done off-line, thereby increasing cost and cycle time.

20 Moving the particle from the first sample surface to a more controlled
21 environment for testing can dramatically improve the chance of success and throughput
22 for elemental identification with either EDS or Auger analysis. A critical part of this
23 process is the strategy for moving the particle. This disclosure describes a novel method
24 for removing a particle of interest from a sample surface, transporting that particle to a
25 second sample surface with a controlled X-ray or Auger background, and performing
26 electron beam-induced X-ray analysis or Auger electron analysis there, using any of the
27 methods discussed above. This eliminates the requirement that the analyzing technique
28 have high spatial resolution, although a technique with high spatial resolution, such as
29 EDS analysis in the SEM and SAM analysis, is generally preferred. For example,
30 techniques without high spatial resolution that could be successfully applied to the
31 situation of a particle on a reduced or non-interfering background include X-ray
32 Photoelectron Spectroscopy (XPS) and X-ray Fluorescence analysis (XRF), which may
33 offer an advantage in unique and specific situations.

The proposed method for particle manipulation and EDS X-ray analysis can be done in-line on existing wafer-manufacturing tools. An in-line procedure using existing manufacturing and inspection tools represents a significant reduction in cycle time for contamination removal. SEM is a routine method for wafer inspection, and analytical methods using the electron beam in an SEM system provide a substantial throughput advantage over the off-line strategies.

Although this disclosure primarily illustrates the use of the novel technique to manipulate and examine particles that are contaminants in the context of semiconductor manufacturing, the reader should note that the term "particle" may be taken to include objects that may not be contaminants in other environments, such as chemical deposits, biological material, or micro-mechanical machines. In the latter cases, the novel methods of manipulation described in this application may be applied to manipulate these objects generally, for purposes other than electron-beam X-ray analysis or Auger electron analysis.

## DRAWINGS

Figure 1 shows the steps of attaching a particle to a micro-manipulator probe and removing the particle to a second surface for analysis.

Figure 2 shows three other methods of attaching a particle to a micro-manipulator probe.

Figure 3 shows the process of modifying electrostatic forces by bombardment with polarizable molecules.

Figure 4 shows the method of simultaneously viewing a particle and modifying the charge state of the particle.

Figure 5 shows several methods for fixing a particle to a second surface for analysis.

Figure 6 shows the analysis of a particle while the particle is fixed to the tip of a micro-manipulator probe.

Figure 7 shows the process of analyzing the composition of a particle removed to a second surface for analysis.

## SUMMARY

We disclose a method for analyzing the composition of a microscopic particle resting on a first sample surface. Usually, the particle will be a contaminant in a semiconductor processing system, although the method is not limited to those

1 circumstances. The method comprises positioning a micro-manipulator probe near the
2 particle; attaching the particle to the probe; moving the probe and the attached particle
3 away from the first sample surface; positioning the particle on a second sample surface;
4 and, analyzing the composition of the particle on the second sample surface by energy-
5 dispersive X-ray analysis, Auger microprobe analysis or any other suitable analytical
6 technique. The second surface has a reduced or non-interfering background signal
7 during analysis, relative to the background signal of the first surface. (We call such a
8 reduced or non-interfering background signal a "controlled" background signal in the
9 claims.) We also disclose methods for adjusting the electrostatic forces and DC
10 potentials between the probe, the particle, and the sample surfaces to effect removal of
11 the particle, and its transfer and relocation to the second sample surface. These include
12 adjusting electrostatic forces to create an attractive force between the probe and particle.
13 Adjustment of the electrostatic forces may include locally adjusting the energy or
14 intensity (intensity means beam current for electron and ion beams) of an electron beam,
15 ion beam or photon beam incident on the individual components of the sample system,
16 which includes the probe tip, particle and first sample surface, to create an electrostatic
17 attraction between the particle and probe tip, or an electrostatic repulsion between the
18 particle and the first sample surface. This procedure is reversed to transfer the particle
19 from the probe tip to the second sample surface.

20 The second sample surface may be the probe tip itself. In this case the probe tip
21 is composed of a controlled background material. Due to the possibility of transmission
22 of the energetic beam through a tiny particle, or scattering of the energetic beam onto the
23 underlying surface, it may be necessary to translate the probe tip with the particle
24 attached over a surface composed of a controlled background material, or alternatively
25 translate such a controlled background surface beneath the probe tip with the particle
26 attached. In this description, "under" and "beneath" refer to the side of the particle
27 opposite the side on which the energetic beam is incident (i.e.: the transmitted side).

28 **DETAILED DESCRIPTION**

29 The analysis of microscopic particles, particularly in semiconductor
30 manufacturing, is typically done inside a Scanning Electron Microscope (SEM), Focused
31 Ion Beam (FIB) instrument, or Scanning Auger Microprobe (SAM). The FIB instrument
32 may be either a single-beam model, or a dual-beam (both SEM and ion beam) model.
33 Typical FIB instruments are those manufactured by FEI Company of Hillsboro, Oregon,

1 as models 200, 235, 820, 830, or 835. The probe (120) referred to below is a
2 component of a micro-manipulator tool attached to the FIB instrument with vacuum
3 feed-through. A typical such micro-manipulator tool is the Model 100 manufactured by
4 Omniprobe, Inc. of Dallas, Texas. Typical SAM instruments include the JAMP-7810
5 and JAMP-7830F manufactured by JEOL USA, Inc. of Peabody, Massachusetts.

6 Figure 1 depicts the general setup for particle manipulation and analysis. Fig. 1A
7 shows a particle (100) of interest resting on a first sample surface (110). A micro-
8 manipulator probe (120) is positioned near the particle (100). The probe tip can be
9 electrostatically charged relative to the particle and the first sample surface.
10 Alternatively, a voltage source (130) may be connected between the probe (120) and the
11 first sample surface (110). The local electrostatic charge on the particle can be modified
12 by the irradiation of the particle by a charged particle beam. Figs. 1B through 1D show,
13 respectively, the irradiation of the particle (100) and first sample surface (110) by
14 photons or a charged-particle beam (140) to cause attachment of the particle (100) to the
15 probe (120), the removal of the probe (120) and attached particle (100) from the first
16 sample surface (110), and the deposition of the particle (100) on a second sample surface
17 (150) for analysis. The drawings are not to scale.

18 Attaching the particle to the probe

19 Strong electrostatic forces exist on particles in a vacuum. The presence of static
20 charges on the particle (100) and the probe (120) leads to the creation of image charges
21 on the opposite surfaces. These image charges create forces that are proportional to the
22 area exposed and inversely proportional to the distance between the objects. Reducing
23 or increasing the exposed area will therefore either reduce or increase the force acting on
24 the particle (100), and the resultant adhesion between probe (120) and particle (100).
25 This can be used as a straightforward method to remove particles of interest from the
26 sample, using either a conducting or insulating probe (120). Conducting probes allow
27 more versatility through the introduction of static or time varying voltages or
28 electrostatic charges to the probe (120) from a voltage or electrostatic charge source
29 (130), as shown generally in Fig. 1A.

30 The shape of the tip of the probe (120) will also influence the electric fields at the
31 tip. Static electric charges on a blunt tip will exert stronger influence on a particle in line
32 with the tip than a sharply pointed tip. In contrast, in the case of a DC potential on a
33 conductive tip, a sharp tip will produce the strongest field concentration at the tip. The

1 probe (120) can be moved into proximity to the particle (100) while imaging with, for
2 example, the electron beam (140) available in the FIB instrument, as shown in Fig. 1B.
3 The electron beam will also affect the charge distribution in the surface-particle-probe
4 system, and thus can assist attraction of the particle (100) to the probe (120). An
5 application of this effect is discussed below. The electron beam (140) depicted in Fig.
6 1B and other drawings should be understood to also be a charged-particle beam or
7 photon beam generally, and may, for example, consist of an ion beam. These, and beams
8 of photons, such as from a laser, are referred to collectively in the claims as "energetic"
9 beams.

10 In general, the adjustment of electrostatic forces on the system may comprise
11 adjusting the energy of an electron beam (140) incident on the particle (100), probe
12 (120), and first sample surface (110) to create a relative electrostatic attraction between
13 the particle (100) and the probe (120), and a relative electrostatic repulsion between the
14 particle (100) and the first sample surface (110). The process may be assisted by a
15 voltage source (130) connected between the first sample surface (110) and the probe
16 (120). Clearly, the impinging beam (140) could also be a beam of photons, having
17 sufficient energy to release photoelectrons, which thus change the charge distribution in
18 the system, and the electrostatic forces involved.

19 The preferred embodiment may also be carried out using an adhesive (160) on the
20 probe (120), as shown in Fig. 2A. An acceptable adhesive (160) could be any having a
21 low vapor pressure, such as vacuum grease, low melting point waxes, or other low vapor
22 pressure glues. In this case, the forces of adhesion simply capture the particle (100),
23 notwithstanding existing electrostatic forces.

24 In another embodiment, shown in Fig. 2B, tweezers (170) connected to the probe
25 (120) grasp the particle (100) and remove it from the first sample surface (110). Suitable
26 device having tweezers (170) or similar grippers are those manufactured by MEMS
27 Precision Instruments in Berkeley, CA.

28 The probe (120) can touch the particle (100), but this is not necessary in many
29 cases, as the particle (100) will jump to the probe (120) due to the electrostatic
30 attraction. The electrostatic field is controlled by surface area and therefore enhanced
31 with a blunt tip on the probe (120), or the blunt side of a particle (100) or the probe
32 (120), whereas DC potentials are enhanced by a pointed tip that concentrates the field
33 lines. Figs. 2C and 2D show examples of strategies for particle (100) attachment and

1 transfer by controlling the surface area of the particle (100) exposed to the manipulator,
2 by applying the tip (125) of the probe (120) and the side (135) of the probe (120) to the
3 particle to achieve the desired movement of the particle (100).

4 An additional method of adjusting the electrostatic fields in the particle-probe-
5 surface system, for both attaching and removing the particle (100) comprises depositing
6 a conductive material on the first sample surface (110) or second sample surface (150),
7 as the case may be, to distribute and modify the electrostatic charge on the surface at the
8 location of the particle to create either an attractive or a repulsive force on the particle,
9 as desired. Figure 3A depicts the deposit of polarizable molecules (250), such as water,
10 on the sample surface (110). Figure 3B depicts the deposit of a conductive film (255) by
11 evaporation of a source. Figure 3C depicts a directed jet (240) of gas (245) applied to a
12 surface (110) having a particle (100) resting upon the surface (110). The gas (245) is
13 decomposed by an energetic beam (140), which may be an electron beam, an ion beam,
14 or photons, such as from a laser.

15 A method of simultaneously viewing a particle (100) in a vacuum system and
16 adjusting the charge state of the particle is shown in Figure 4. The SEM beam and the
17 ion beam in typical FIB instruments are scanned over the object of interest in a raster
18 pattern (260). This scanning, synchronized with emitted secondary electrons, generates
19 the electrical signal that is displayed as an image to the operator of the instrument. Since
20 the scanning beam necessarily comprises charged particles, and causes charged particles,
21 such as secondary electrons, to be emitted from the sample, it may itself be used to
22 change the charge state of the particle (100). FIB instruments typically use digital scan
23 generators that digitally increment the position of the beam spot through a raster pattern,
24 one line at a time, often reversing direction between lines to eliminate the flyback after
25 each line that characterizes traditional analog scanners. So the operator, or the computer
26 program controlling the scan, can determine the dwell time on a per-pixel basis. For
27 example, a box covering the particle (or the exact outline shape of the particle) can be
28 programmed with zero dwell time, and therefore blanked during the scan. Any dwell
29 time can be set up to the maximum time allowed by the line rate to avoid image
30 distortion in a single scan. It is also possible to alternately scan around the box, and then
31 scan in the box with different parameters, and do this so quickly that the human eye
32 would not see an interruption.

1 Fig. 4 shows the steps of rastering a primary electron beam (270) over a field of
2 view that includes the particle (100); generating and detecting secondary electrons (280)
3 that are synchronized with the primary beam (270); and modifying the raster scan pattern
4 (260) to specify dwell time and location for specific pixels in the field of the raster (260)
5 associated with the particle (100) to be incorporated and added to the standard raster
6 pattern. The particle (100) then experiences an excess or a reduction of negative charge
7 relative to the sample surface (150) under the rest of the raster (260). Thus the
8 electrostatic field between the particle (100) and the probe (120) and sample surface
9 (150) can be adjusted to achieve attraction or repulsion, as desired. The raster may be
10 generated by ion beams as well, and in the same fashion, by a scanning laser.

11 Transferring the particle

12 Once the particle (100) is attached to the probe (120) by any of the means just
13 described, the probe (120) can be moved within the vacuum environment either manually
14 or via automated probe (120) hardware. An alternative method would be to raise or
15 retract the probe (120) slightly and move the sample stage to bring a controlled
16 background material under the probe (120).

17 The particle (100) can also be transferred by the probe (120) to the second
18 sample surface (150) consisting substantially of a controlled background material having
19 a low background or non-interfering background signal. For analysis by EDS, low
20 atomic-number materials such as carbon or beryllium produce low-energy X-rays that
21 will not interfere with most non-organic particle-analysis processes. An atomic number
22 less than or equal to 12 is preferred. Organic particles will obviously require a non-
23 organic background material. Examples of the low-background materials for the second
24 sample surface (150) include photoresist, carbon planchette, beryllium foil, conductive
25 carbon-based paste (colloidal graphite suspensions), polymer membranes, or carbon
26 nanotube needles. Any material whose X-ray background does not interfere with the
27 typical materials in the fabrication process may be acceptable for the second sample
28 surface (150). In some cases, the second sample surface (150) may be a different part of
29 the first sample surface (110). In other cases, where the composition of the particle
30 (100) is partly known or suspected, the material of the second sample surface (150)
31 should have a background signal different that the signals expected from the particle
32 (100). Care must be taken that the choice of the second sample surface (150) does not
33 obscure possible signals from contaminants from outside the fabrication facility, such as

1 impurities in incoming gases or chemicals. For Auger analysis of the particle on the
2 second surface, the second surface should consist of low Auger electron background or
3 non-interfering Auger electron background. The composition of the second surface
4 should be consistent to a depth greater than that of any depth profiling that will be
5 performed on the particle. It will be helpful, but not necessary for the second surface
6 material to be electrically and thermally conductive to minimize any charging or thermo-
7 mechanical drift problems associated with high incident electron beam currents. A pre-
8 sputtering of the second surface, before transfer of the particle will remove any native
9 surface coating (mostly carbon and oxygen) and simplify the analysis. This pre-
10 sputtering can be performed, for example, with the depth profiling ion source in the
11 Auger, or the ion beam in the FIB. That the composition of the second surface is well
12 known eliminates the need to acquire background analyses which improves throughput.

13 Figure 5 shows several methods for transferring the attached particle (100) from
14 the probe (120) to the second sample surface (150) for the analysis. Fig. 5A shows the
15 particle suspended on an underlying framework (190), thin relative to the penetration
16 depth of the analysis beam (140). The framework (190) would typically be a TEM grid,
17 possibly having a polymer membrane (195) such as FORMVAR across the grid
18 openings.

19 Fig. 5B shows the particle attached to the second sample surface (150) by an
20 adhesive (200) on the second sample surface (150). Fig. 5C shows a second sample
21 surface (150) comprising a background material (210) having a low modulus of
22 elasticity, such as vacuum grease, low-melting point wax, or low-modulus polymer. In
23 this case the particle (100) can be pushed into the low-modulus material (210) and stuck
24 there.

25 Fig. 5D shows a wrinkled surface (220) on an insulating second sample surface
26 (150). The wrinkled surface (220) allows an increased area of contact between the
27 particle (100) and the second sample surface (150), thus changing the electrostatic forces
28 between them.

29 Fig. 5E shows an electrified pattern (230) written on the second sample surface
30 (150) by the charged-particle beam (140). The electrostatic field of such a pattern can
31 assist in the transfer of the particle from the probe (120) to the second sample surface
32 (150).

1 Figure 5F shows a porous second sample surface (150) having holes or pores
2 (290). Such surfaces may be micro-pore filters, such as the MICROPORE series of
3 filters manufactured by 3M Corporation of St. Paul, Minnesota, glass fiber filters such as
4 the FILTRETE or EMPORE series of filters manufactured by 3M Corporation of St.
5 Paul, Minnesota, or "holey carbon" films, such as the QUANTIFOIL series manufactured
6 by Structure Probe, Inc. of West Chester, PA. These surfaces have the advantage that
7 particles (100) will rest or be electrostatically captured in the holes or pores and be held
8 there for analysis.

9 In some cases it may be necessary to search for areas of high local static fields
10 sufficient to remove the particle (100) from the probe (120) without contact (if that is
11 desired).

12 Of course, the methods described in the previous section for adjusting the
13 electrostatic forces in the particle-probe-sample surface system for attaching the particle
14 (100) to the probe (120) can also be used to remove the particle (100) from the probe
15 (120) and attach it to the second sample surface (150). In particular, the voltage or
16 charge source (130) may generate a rapid transient or resonant phenomenon, for
17 example, by rapidly switching stored negative charge from a capacitor through the probe
18 (120), or by a time-varying voltage, such as a square wave or pulse, applied to the probe
19 (120) from the source (130).

20 Analyzing the particle

21 X-ray analysis or Auger analysis can be performed with the particle (100) directly
22 on the probe tip (125), as shown in Fig. 6. This will of course result in X-ray or Auger
23 electron generation from the probe tip (120) itself. Other interfering signals can be
24 reduced by either using a low-background or non-interfering background material for the
25 probe tip material, as discussed above, placing a low-background or non-interfering
26 background material under the probe (120) during this analysis, or by dropping the stage
27 and all other hardware from near the probe (120). Removal of the particle (100) after
28 this step can be performed destructively since the particle (100) analysis has already been
29 done. Example destructive methods might include inserting the probe (120) in a plasma
30 cleaner of some kind, rubbing the particle (100) off on a mechanical transfer structure
31 such as a V-groove, irradiating the probe optically either in vacuum or after exposure to
32 the atmosphere, or ablating the particle (100).

1 Usually, however, the particle (100) will be analyzed on a second sample surface
2 (150), as depicted generally in Fig. 7, where the particle (100) is irradiated with a
3 charged-particle analysis beam (140), causing it to emit characteristic Auger electrons or
4 X-rays (180) for compositional analysis, by any of the methods described in the
5 Background section of this application. In the claims, the term "emissions" denotes
6 either Auger electrons or X-rays.

7 Analyzing the particle on the probe tip

8 The second sample (150) surface may be the probe tip (135) itself. In this case
9 the probe tip (135) is composed of a controlled background material. In the case of a
10 analysis instrument such as SAM or FIB in which ion beam milling of the surface is
11 possible, the surface of the probe tip (135) can be ion milled prior to attachment of the
12 particle (100) to the tip (135) to reduce signals from the native surface coating and
13 debris on the probe tip (135) surface. Due to the possibility of transmission of the
14 energetic beam (140) through a tiny particle, or scattering of the energetic beam (140)
15 onto the underlying surface, it may be necessary to translate the probe tip (135) with the
16 particle (100) attached over a surface composed of a controlled background material, or
17 alternatively translate such a controlled background surface beneath the probe tip (135)
18 with the particle (100) attached. In this description, "under" and "beneath" refer to the
19 side of the particle (100) opposite the side on which the energetic beam (140) is incident
20 (i.e.: the transmitted side).

21 Since those skilled in the art can modify the specific embodiments described
22 above, we intend that the claims be interpreted to cover such modifications and
23 equivalents.

24 We claim:

## CLAIMS

1 1. A method for analyzing the composition of a particle; the particle resting on a
2 first sample surface; the method comprising the steps of:
3 positioning a micro-manipulator probe near the particle, the probe having a tip;
4 attaching the particle to the probe tip;
5 moving the probe and the attached particle away from the first sample surface;
6 removing the particle from the probe tip to a second sample surface; and,
7 analyzing the composition of the particle on the second sample surface;
8 where the second sample surface has a controlled background signal during analysis
9 relative to the background signal of the first surface.

1 2. The method of claim 1 carried out in an atmosphere.

1 3. The method of claim 1 carried out in a vacuum.

1 4. The method of claim 1 where the particle is attached, moved, and removed while
2 being irradiated by an electron beam.

1 5. The method of claim 1 where the particle is attached, moved, and removed while
2 being irradiated by an ion beam.

1 6. The method of claim 1 where the particle is attached, moved, and removed while
2 being irradiated by a photon beam.

1 7. The method of claim 1 where the second sample surface is a portion of the first
2 sample surface.

1 8. The method of claim 1 where the step of moving the probe and the attached
2 particle away from the first sample surface comprises:
3 fixing the location of the probe;
4 moving the first sample surface relative to the fixed probe,
5 so as to separate the first sample surface from the probe and the attached particle.

1 9. The method of claim 1 where the second sample surface comprises material
2 having an atomic number less than or equal to 12.

1 10. The method of claim 1 where the second sample surface comprises material
2 having a background signal different than that of the signals expected to be generated by
3 analysis of the particle.

1 11. The method of claim 1 where the step of attaching the particle to the probe tip
2 comprises adjusting electrostatic forces to create an attractive force between the probe
3 and particle.

1 12. The method of claim 11 where the adjustment of electrostatic forces further
2 comprises:
3 adjusting the energy of an energetic beam incident on the particle to
4 electrostatically charge the particle, the first sample surface, and the probe tip so
5 as to create an electrostatic attraction between the particle and the probe tip and
6 to create an electrostatic repulsion between the first sample surface and the
7 particle.

1 13. The method of claim 11 where energetic beam is an electron beam.

1 14. The method of claim 11 where energetic beam is an ion beam.

1 15. The method of claim 11 where the energetic beam comprises photons.

1 16. The method of claim 11 where the adjustment of electrostatic forces further
2 comprises:
3 the particle having an electrostatic charge; and,
4 depositing a conductive material on the first sample surface
5 to distribute and modify the electrostatic charge of the first sample surface at the
6 location of the particle.

1 17. The method of claim 16 where the conductive material deposited on the first
2 sample surface comprises polarizable molecules.

1 18. The method of claim 16 where the conductive material deposited on the first
2 sample surface is an evaporated conductive film.

1 19. The method of claim 16 where the step of depositing a conductive material on the
2 first sample surface comprises bombarding the first sample surface with a directed jet of a
3 gas, and decomposing the gas with an energetic beam.

1 20. The method of claim 19 where the energetic beam is an electron beam.

1 21. The method of claim 19 where the energetic beam is an ion beam.

1 22. The method of claim 19 where the energetic beam comprises photons.

1 23. The method of claim 11 where the adjustment of electrostatic forces further
2 comprises:
3 rastering a energetic beam over a field of view that includes the particle;
4 programming the raster scan to have a pre-determined dwell time and location,
5 where the location includes the location of the particle,
6 so as to impart an electrostatic charge to the particle.

1 24. The method of claim 23 where the energetic beam is an electron beam.

1 25. The method of claim 23 where the energetic beam is an ion beam.

1 26. The method of claim 23 where the energetic beam comprises photons.

1 27. The method of claim 11 where the adjustment of electrostatic forces comprises
2 controlling the surface area of the particle exposed to the probe by applying the tip of the
3 probe or the side of the probe to the particle to achieve attachment of the particle to the
4 probe.

1 28. The method of claim 1 where the step of attaching the particle to the probe tip
2 comprises adjusting a DC bias voltage between the probe and the first sample surface.

1 29. The method of claim 1 where the step of attaching the particle to the probe tip
2 comprises grasping the particle with tweezers.

1 30. The method of claim 1 where the step of attaching the particle to the probe tip
2 comprises the probe tip having an adhesive.

1 31. The method of claim 1 where the step of removing the particle from the probe to
2 the second sample surface comprises adjusting a DC bias voltage between the probe and
3 the second sample surface.

1 32. The method of claim 1 where the step of removing the particle from the probe to
2 the second sample surface comprises applying a time-varying potential to the probe.

1 33. The method of claim 32 where the time-varying potential is a pulse.

1 34. The method of claim 32 where the time-varying potential is generated by rapidly
2 switching stored negative charge from a capacitor through the probe.

1 35. The method of claim 32 where the time-varying potential is a sinusoidal voltage.

1 36. The method of claim 1 where the step of removing the particle from the probe to
2 the second sample surface comprises adjusting electrostatic forces to create a repulsive
3 force between the probe and the particle.

1 37. The method of claim 36 where the adjustment of electrostatic forces further
2 comprises:
3 adjusting the energy of an energetic beam incident on the particle to
4 electrostatically charge the particle, the second sample surface, and the probe tip,
5 so as to create an electrostatic repulsion between the particle and the probe tip

6 and to create an electrostatic attraction between the second sample surface and
7 the particle.

1 38. The method of claim 37 where energetic beam is an electron beam.

1 39. The method of claim 37 where energetic beam is an ion beam.

1 40. The method of claim 37 where the energetic beam comprises photons.

1 41. The method of claim 36 where the adjustment of electrostatic forces further
2 comprises the particle having an electrostatic charge; and, depositing a conductive
3 material on the second sample surface to distribute and modify the charge on the second
4 sample surface at the location of the particle.

1 42. The method of claim 41 where the conductive material deposited on the second
2 sample surface comprises polarizable molecules.

1 43. The method of claim 41 where the conductive material deposited on the second
2 sample surface is an evaporated conductive film.

1 44. The method of claim 41 where the step of depositing a conductive material on the
2 first sample surface comprises bombarding the second sample surface with a directed jet
3 of a gas, and decomposing the gas with an energetic beam.

1 45. The method of claim 44 where the energetic beam is an electron beam.

1 46. The method of claim 44 where the energetic beam is an ion beam.

1 47. The method of claim 44 where the energetic beam comprises photons.

1 48. The method of claim 36 where the adjustment of electrostatic forces further
2 comprises:

3 rastering a energetic beam over a field of view that includes the particle;
4 programming the raster scan to exhibit a pre-determined dwell time and location,
5 where the location includes the location of the particle,
6 so as to impart an electrostatic charge to the particle.

1 49. The method of claim 48 where the energetic beam is an electron beam.

1 50. The method of claim 48 where the energetic beam is an ion beam.

1 51. The method of claim 48 where the energetic beam comprises photons.

1 52. The method of claim 1 where the second sample surface comprises an adhesive,
2 for engaging the particle.

1 53. The method of claim 1 where the second sample surface has an elastic modulus
2 low compared to the compliance of the probe and the elastic modulus of the particle.

1 54. The method of claim 1 where the second sample surface is insulating; the second
2 sample surface having electrified patterns written into it; the charge of the electrified
3 patterns being opposite to that of the particle.

1 55. The method of claim 1 where the second sample surface is wrinkled.

1 56. The method of claim 1 where the step of analyzing the composition of the particle
2 further comprises:
3 irradiating the particle with an analysis beam; and,
4 detecting emissions from the particle.

1

2 57. The method of claim 56 where the analysis beam is an electron beam.

1 58. The method of claim 56 where the analysis beam is an ion beam.

1 59. The method of claim 56 where the analysis beam comprises photons.

1 60. The method of claim 1 where the second sample surface is self-supporting, but is
2 thin relative to the penetration depth of the analysis beam.

1 61. The method of claim 1 where the second sample surface is a porous surface.

1 62. The method of claim 1 where the second sample surface is thin relative to the
2 penetration depth of the analysis beam, and the second sample surface is supported by an
3 underlying framework.

1 63. The method of claim 62 where the underlying framework is a grid.

1 64. The method of claim 1 where the second sample surface is the probe tip; and,
2 where the step of analyzing the composition of the particle comprises analyzing the
3 composition of the particle on the probe tip.

1 65. The method of claim 64 where the probe and the attached particle are moved
2 away from the first sample surface by holding the position of the probe fixed and moving
3 the first sample surface away from the probe and the attached particle.

Fig. 1–A

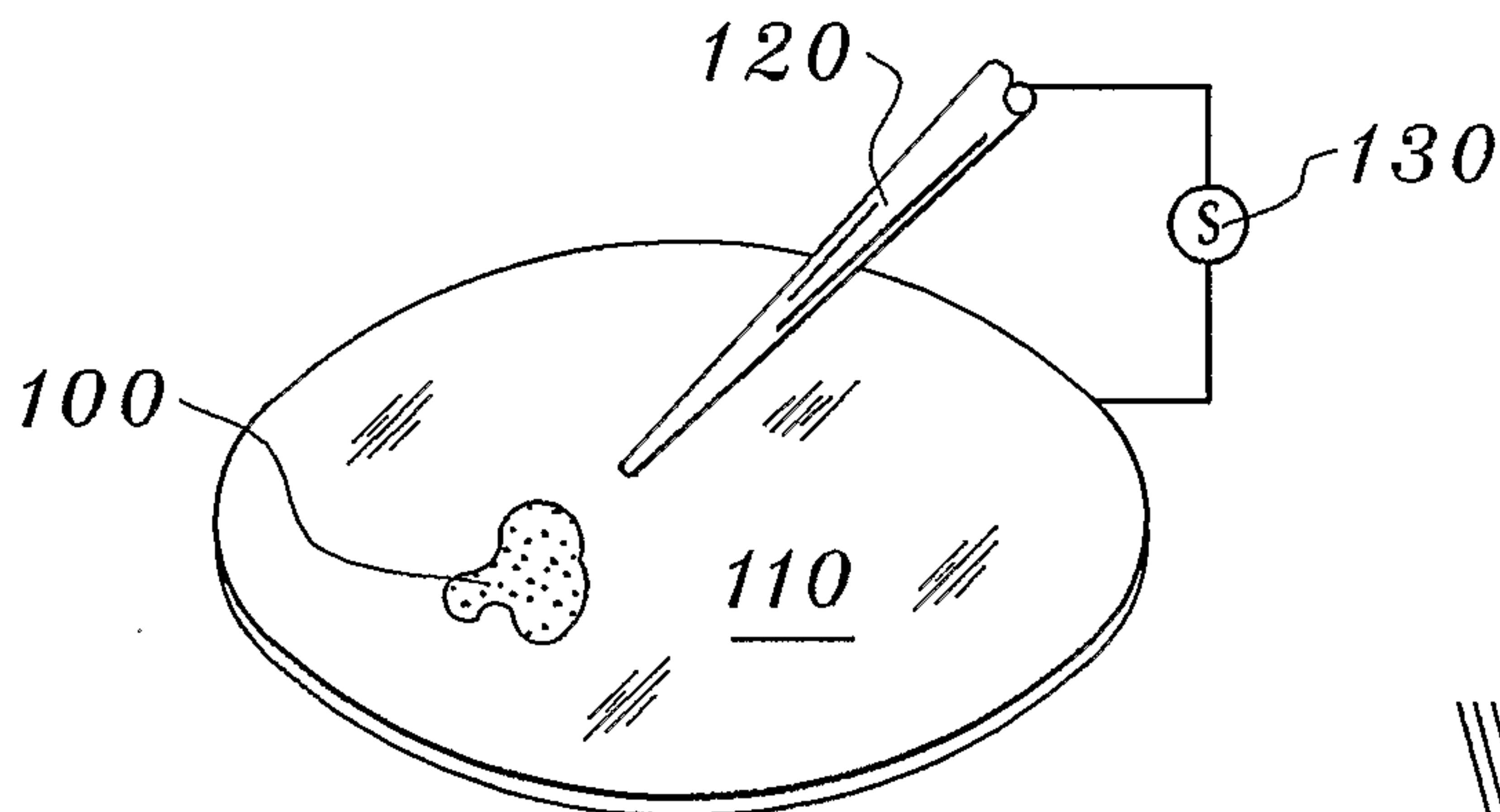



Fig. 1–B

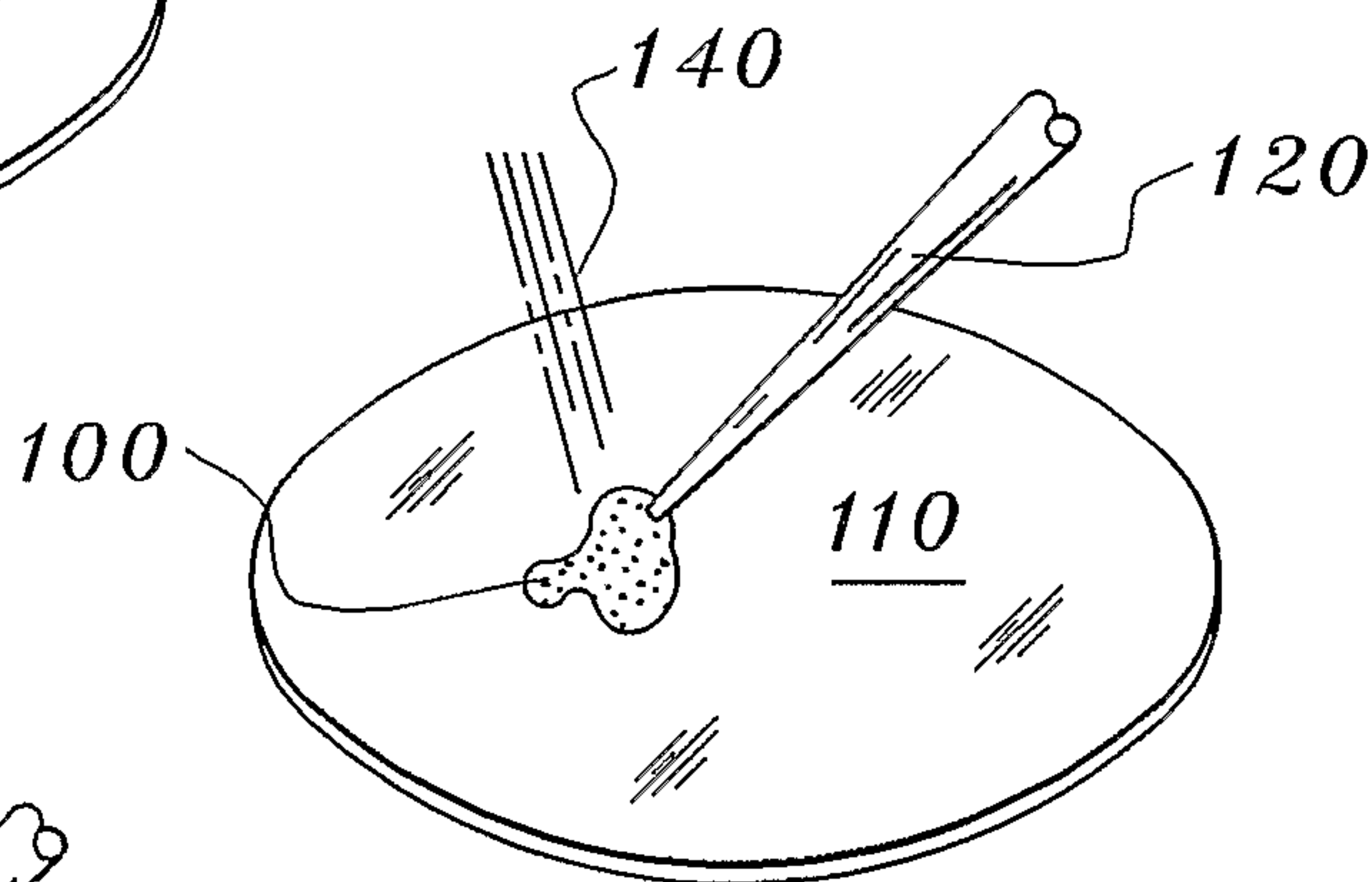
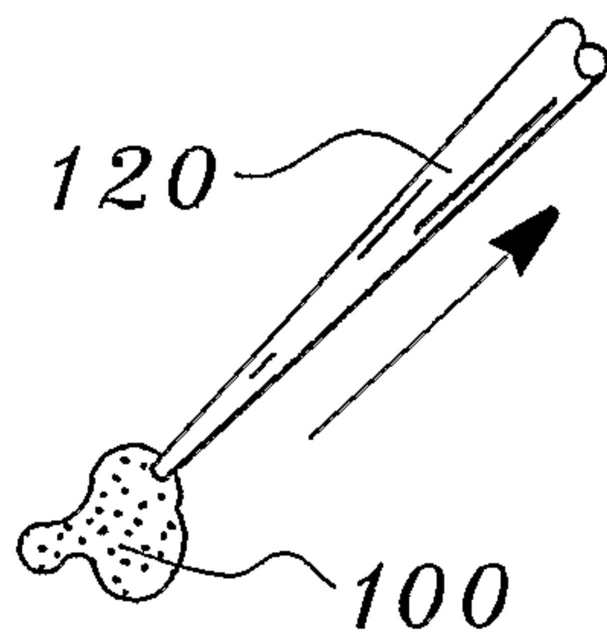



Fig. 1–C



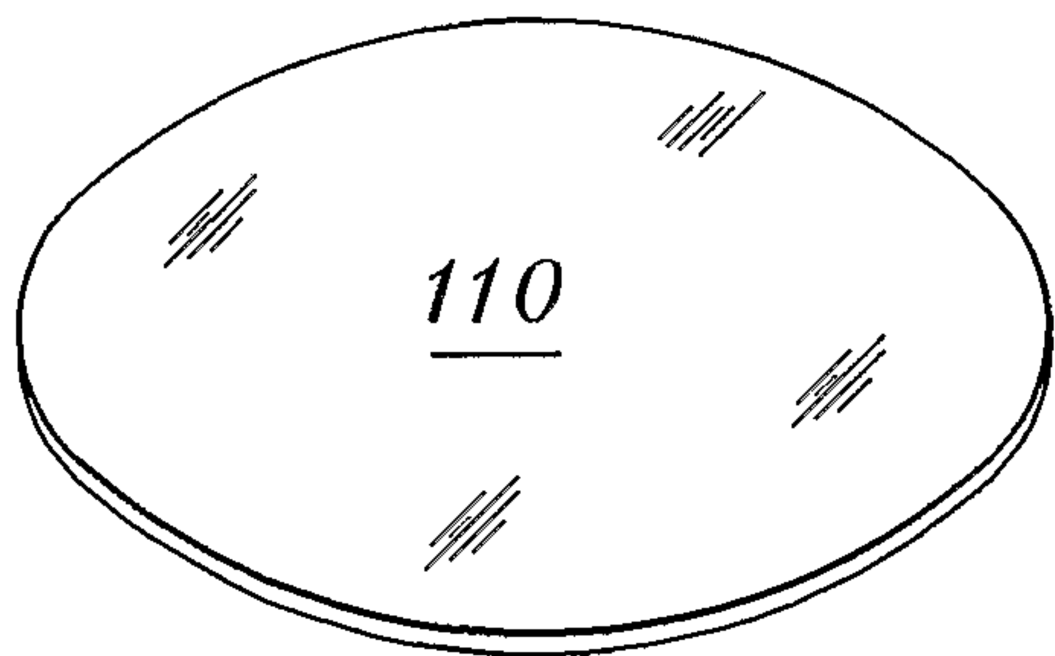




Fig. 1–D

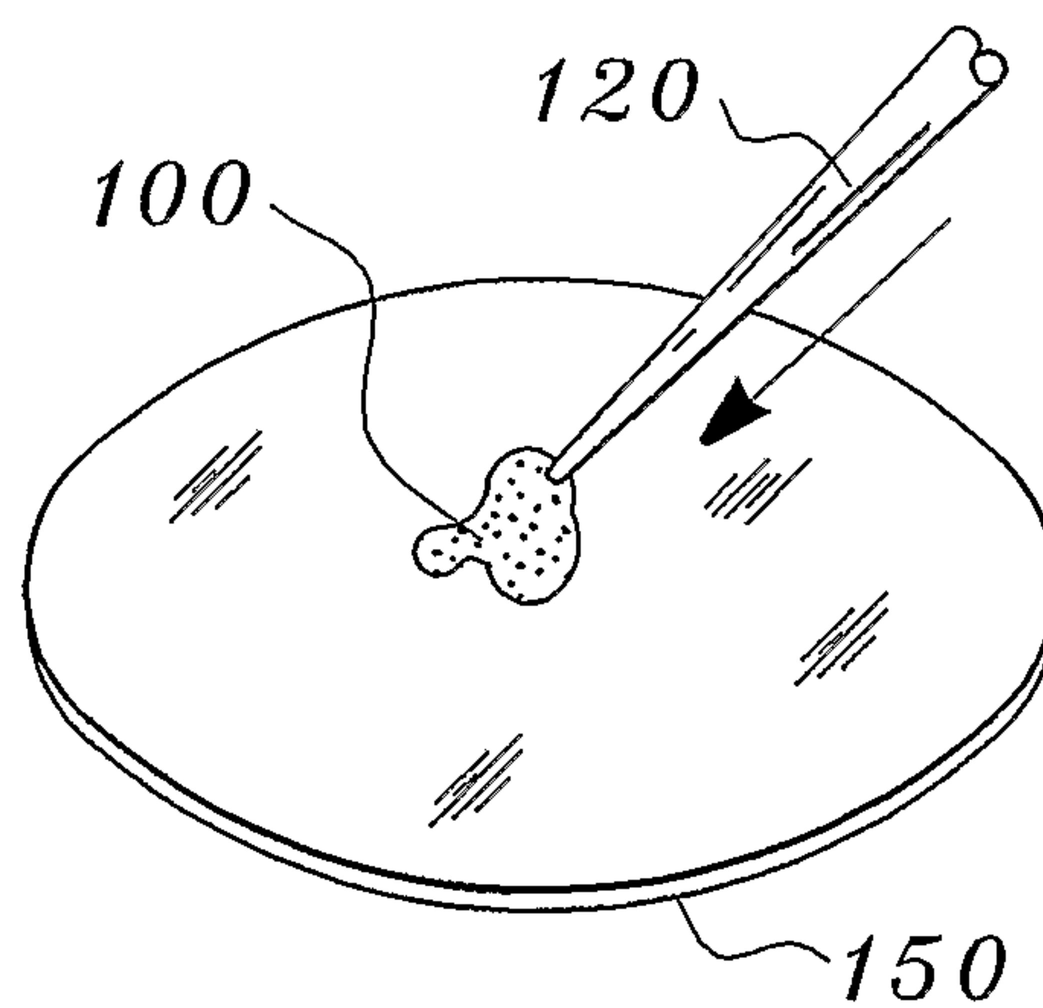



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Fig. 2-A

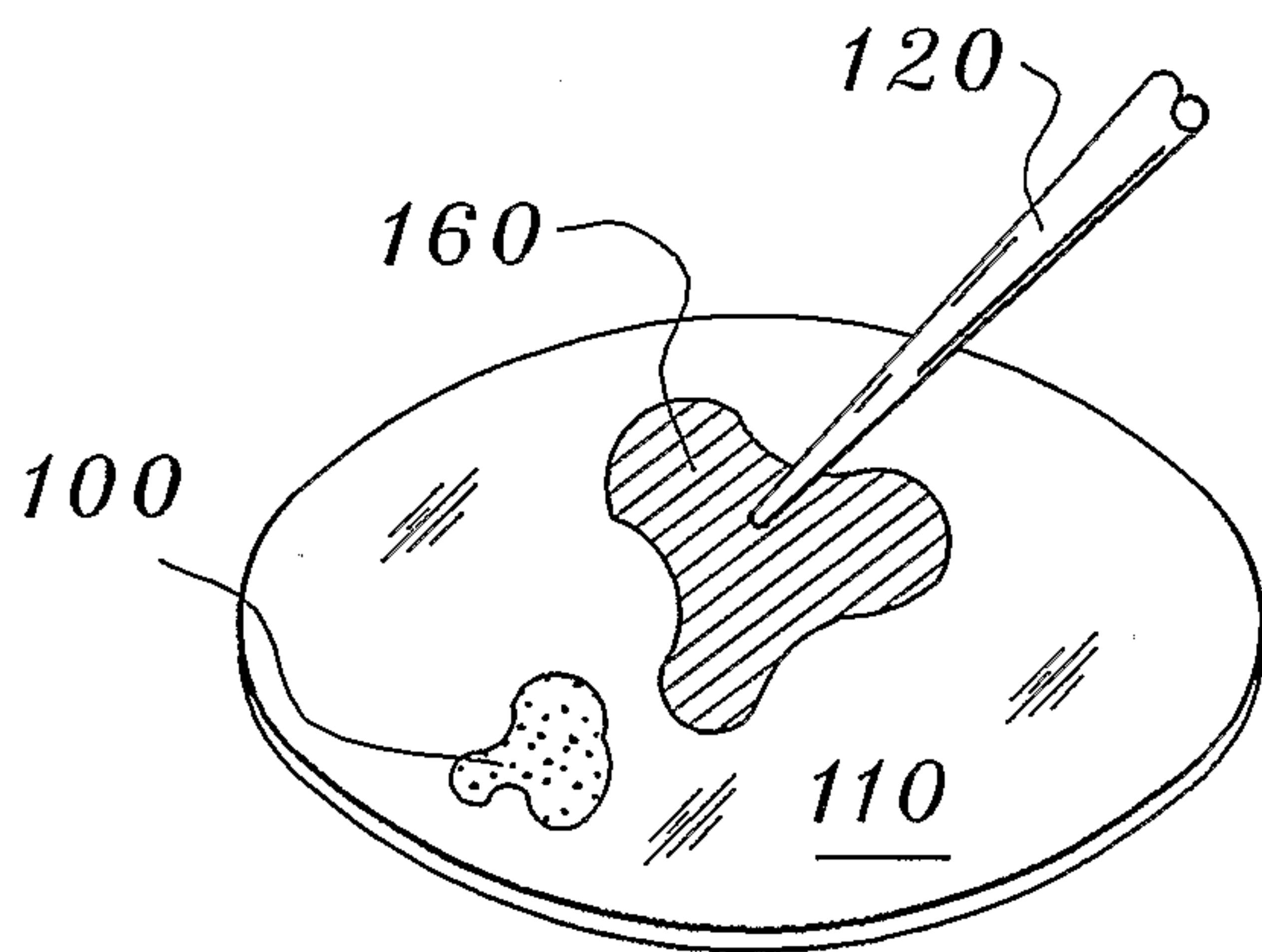
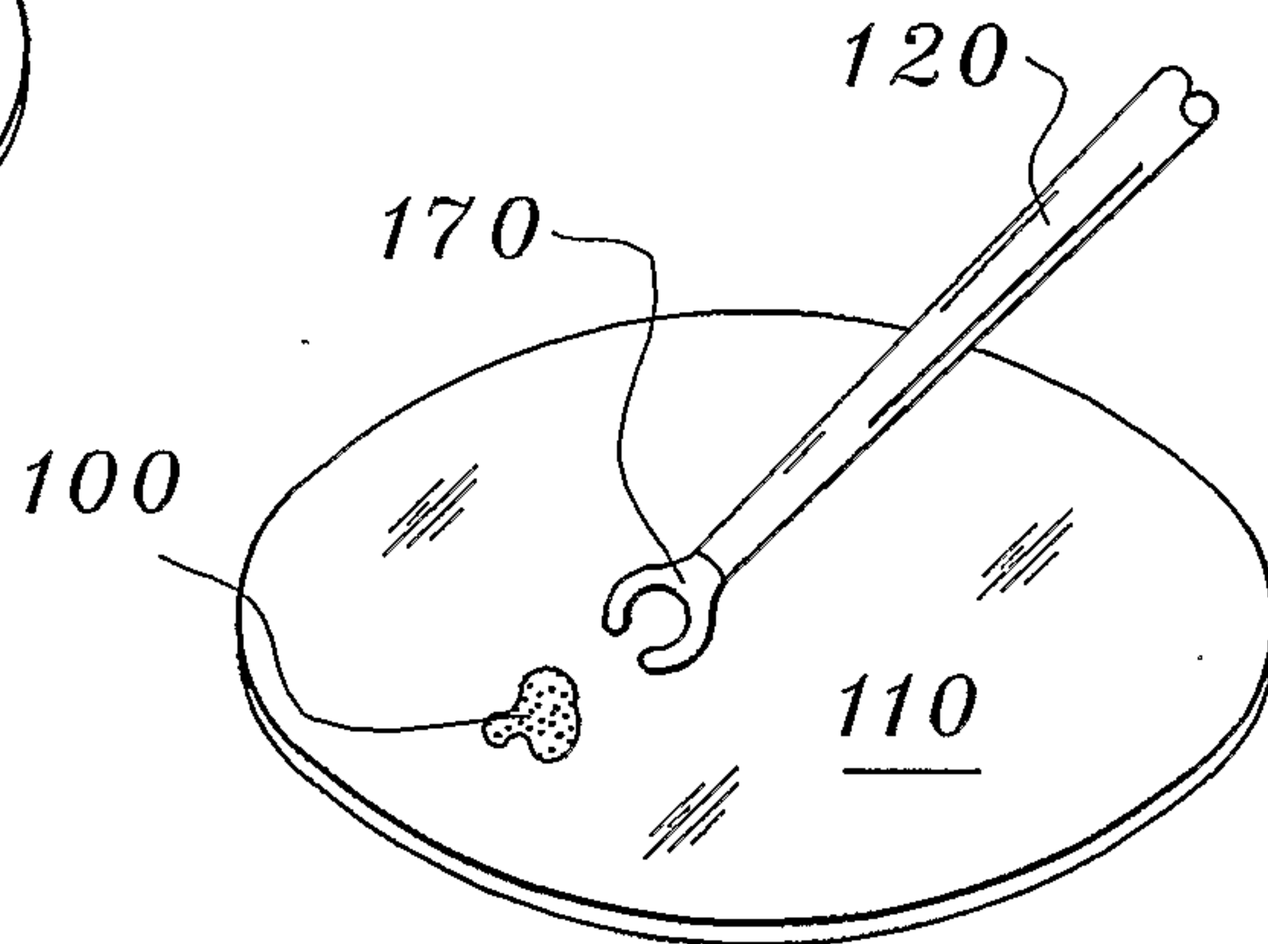



Fig. 2-B

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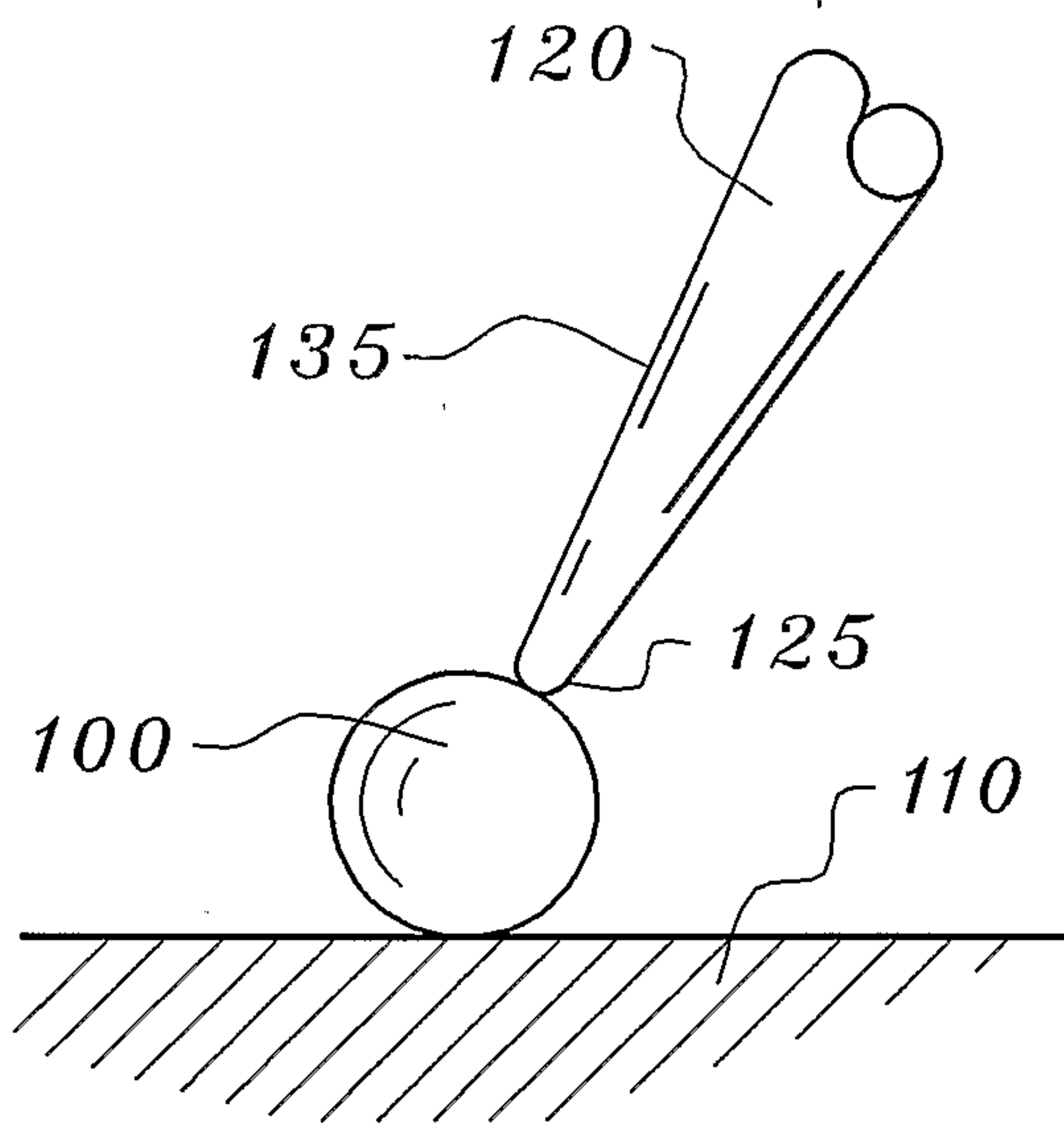



Fig. 2-C

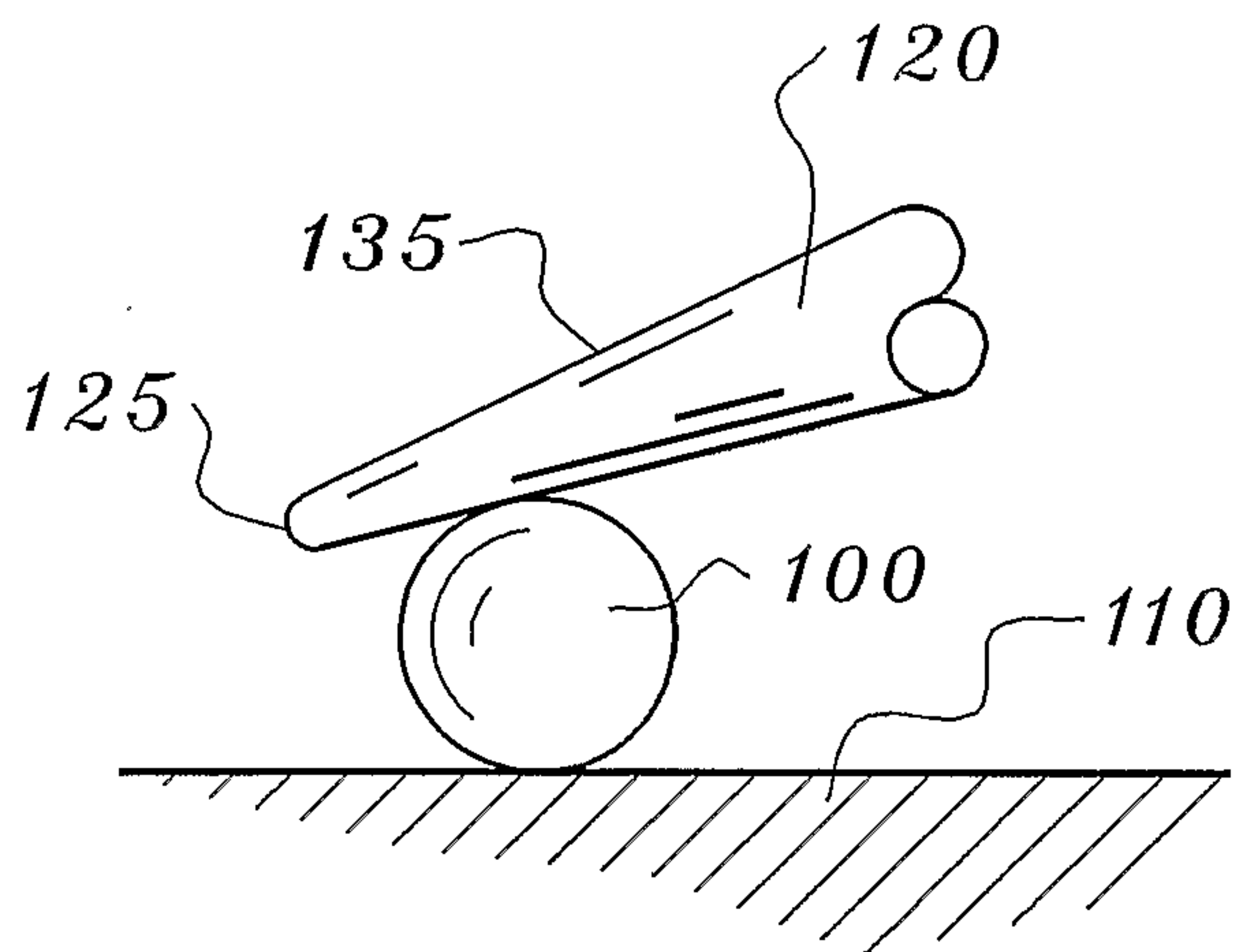



Fig. 2-D

Fig. 3-A

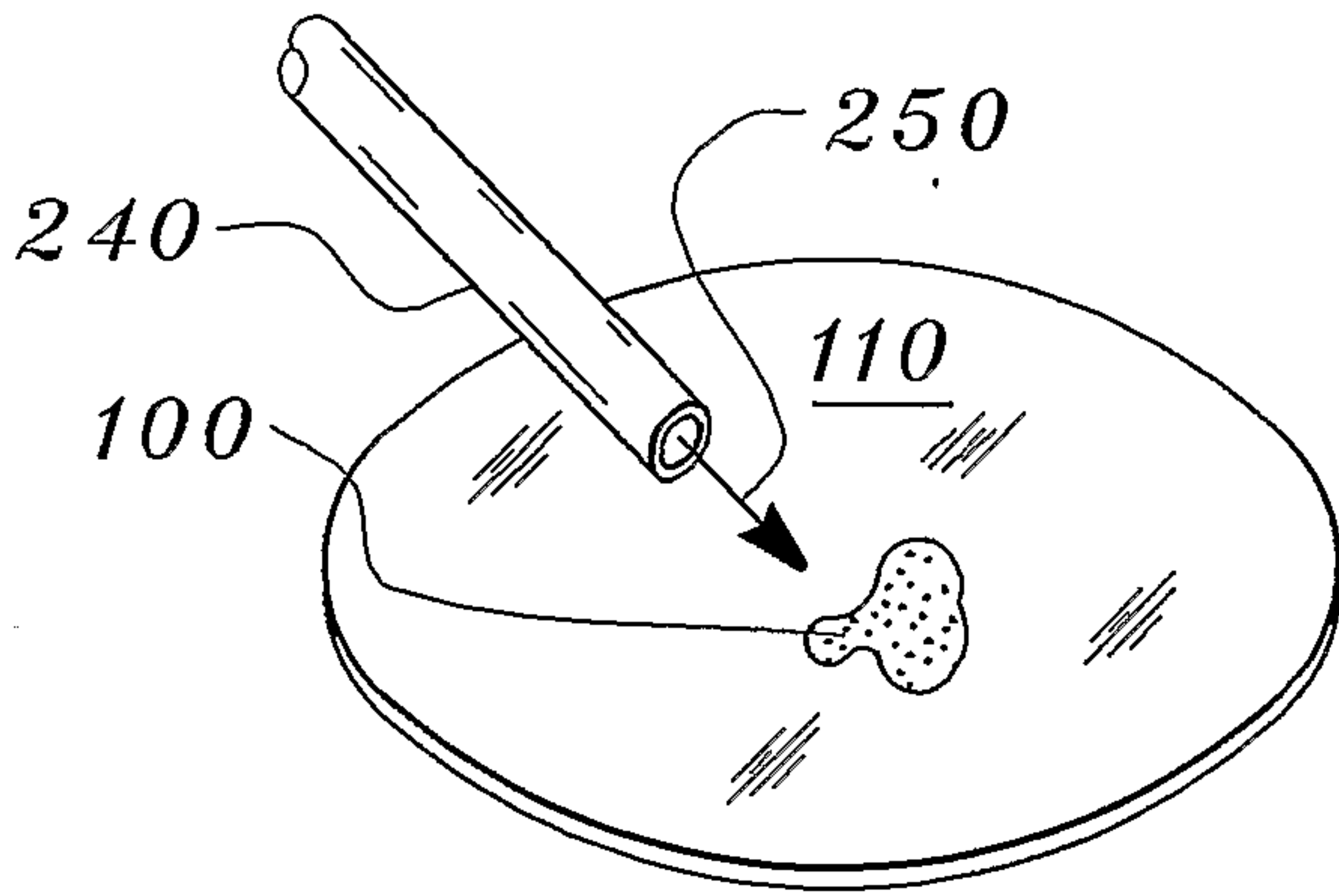
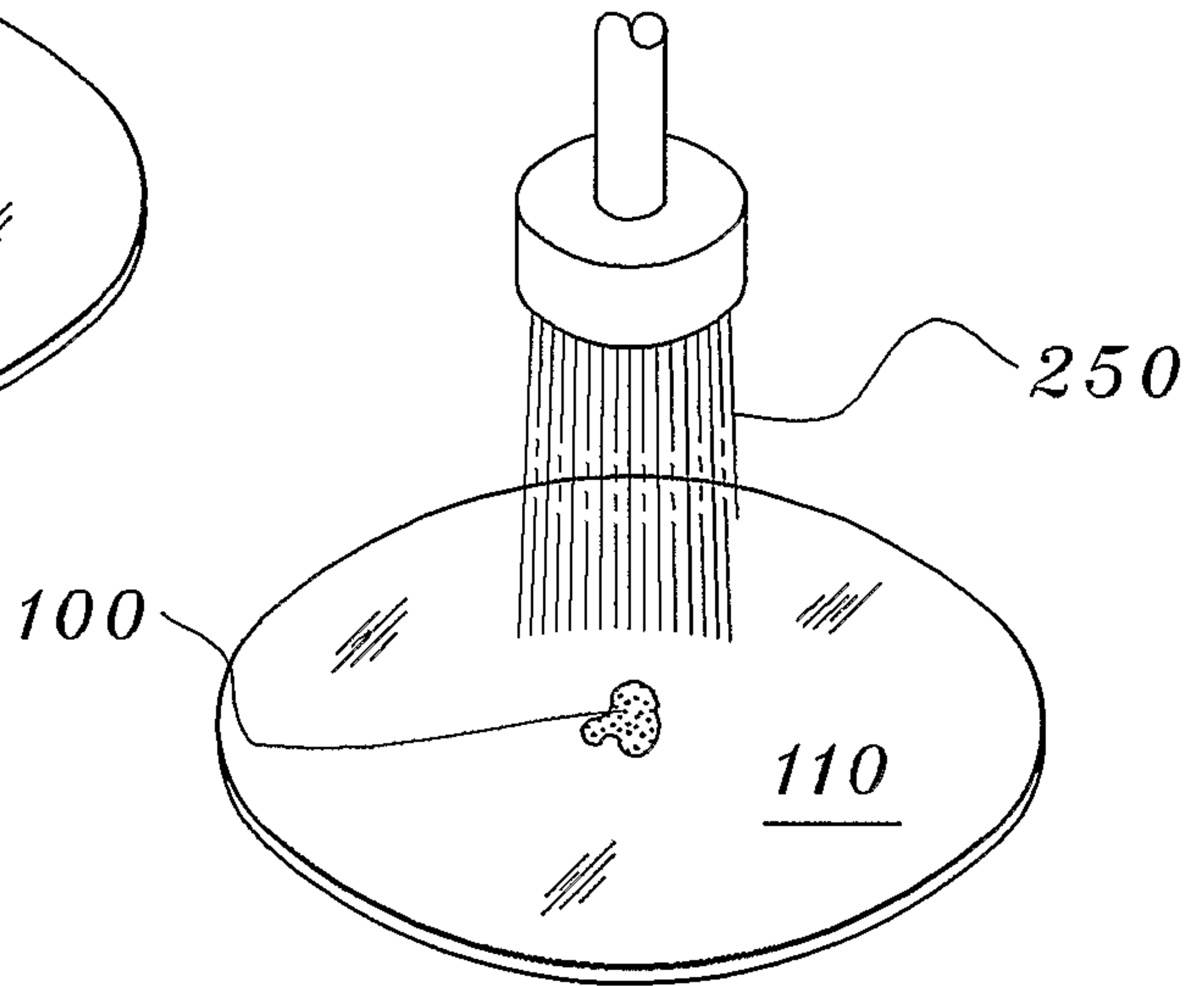



Fig. 3-B

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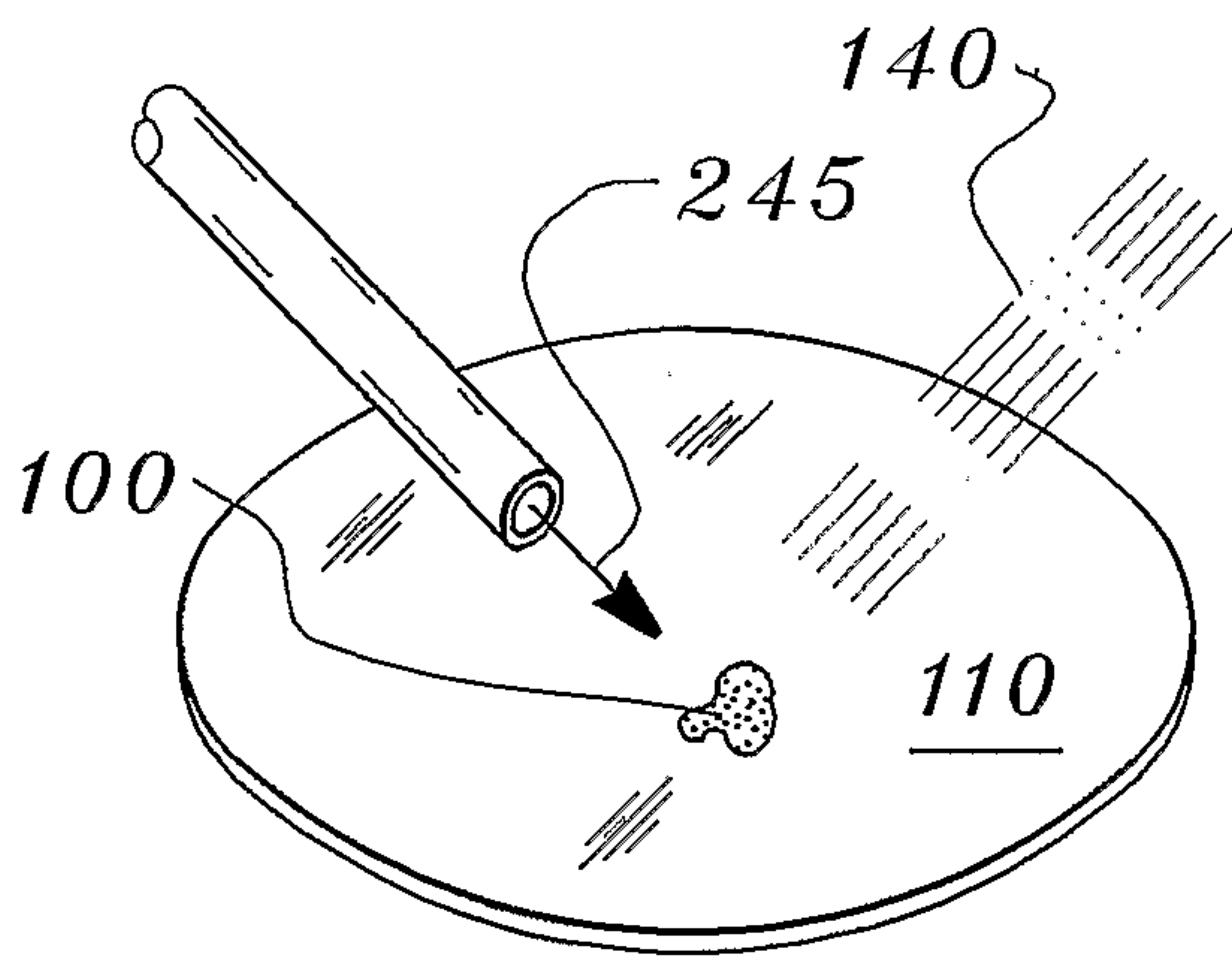


Fig. 3-C

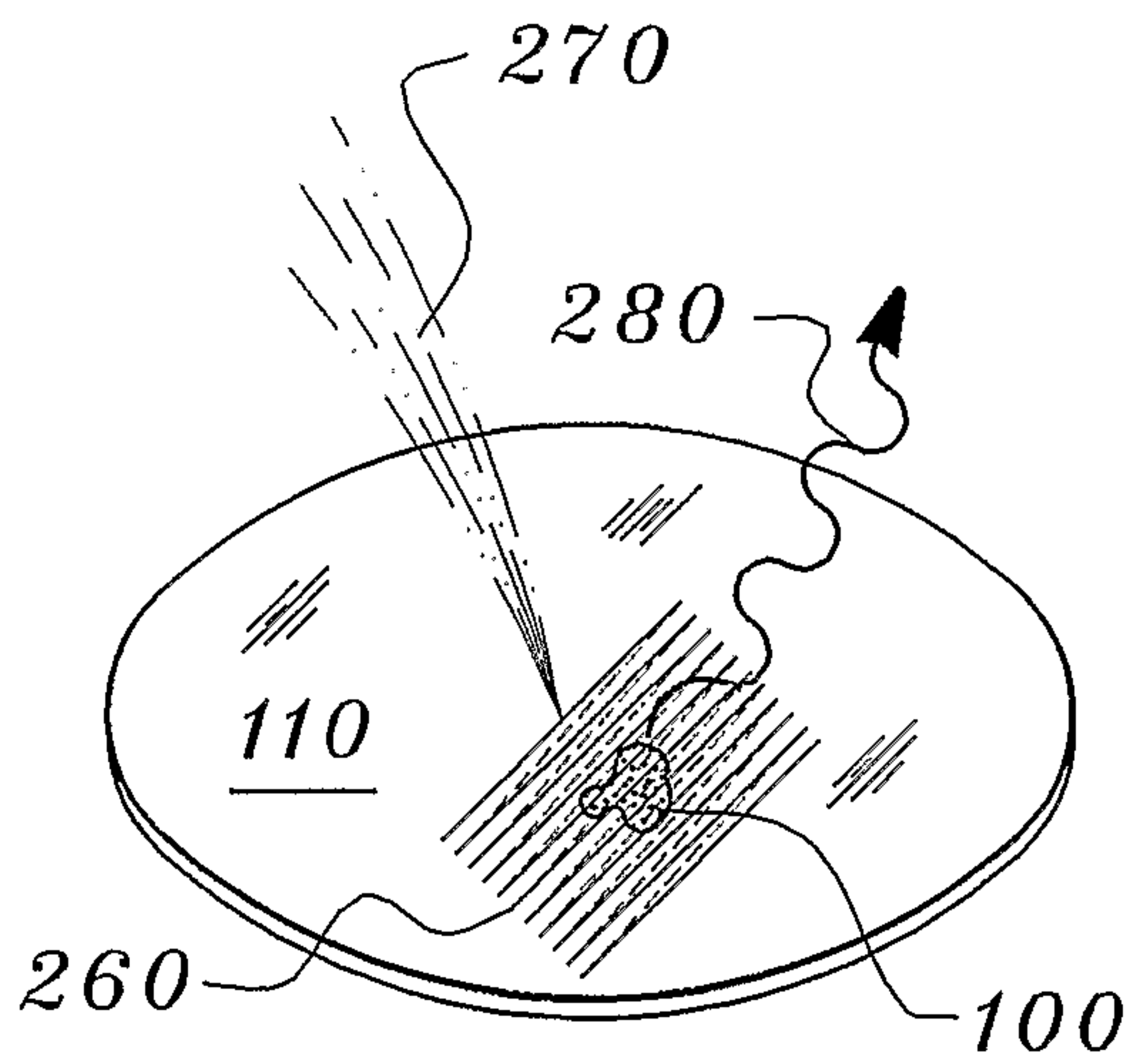


Fig. 4

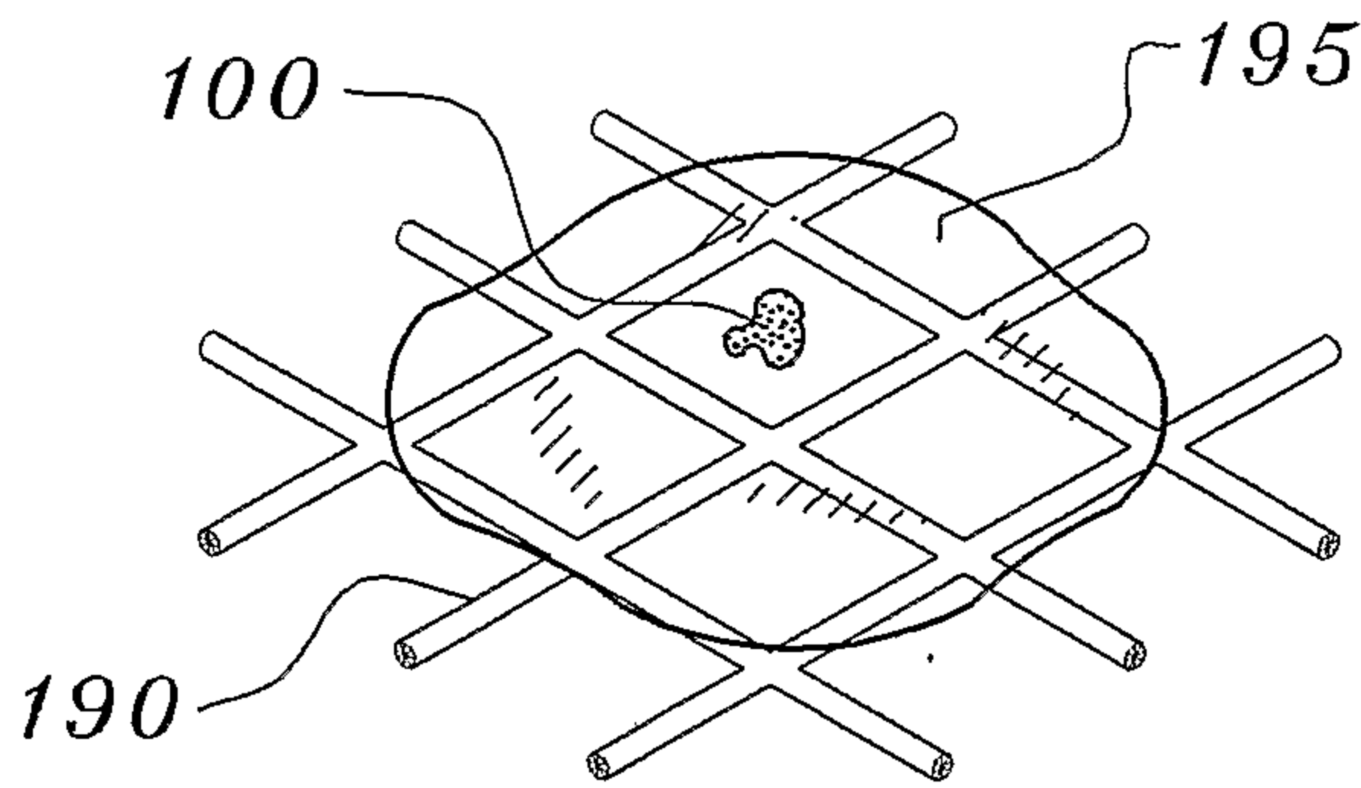


Fig. 5-A

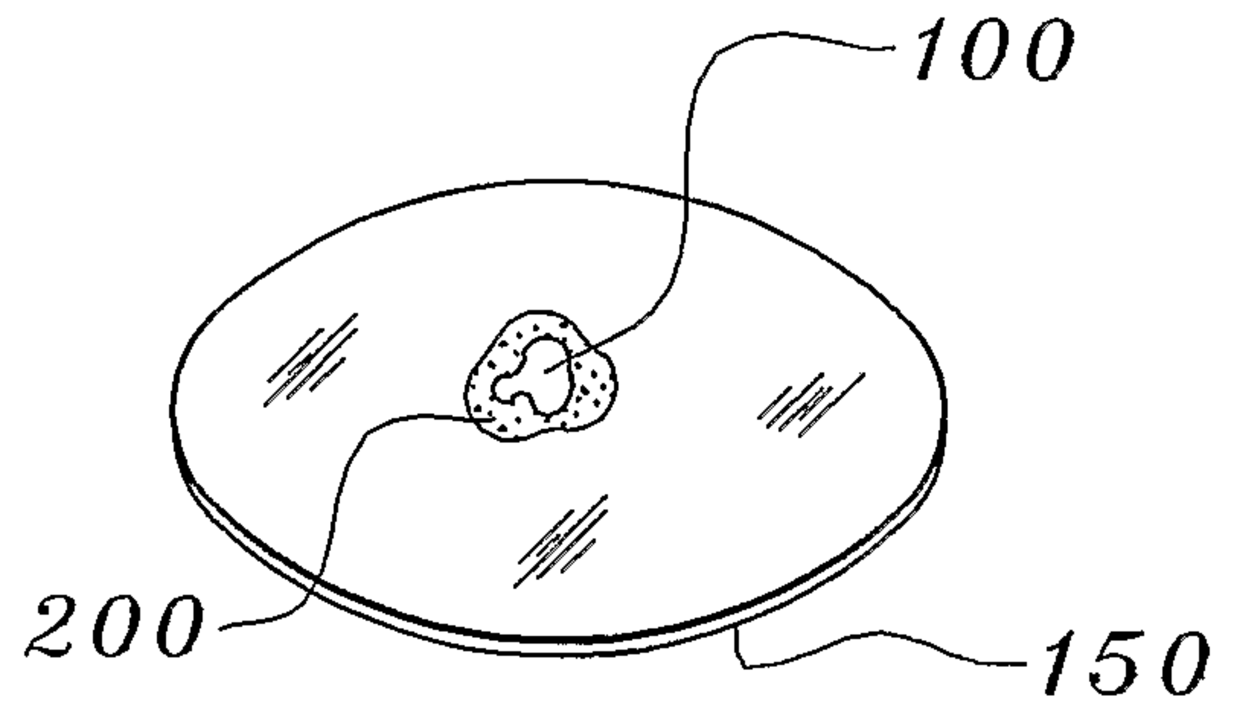


Fig. 5-B

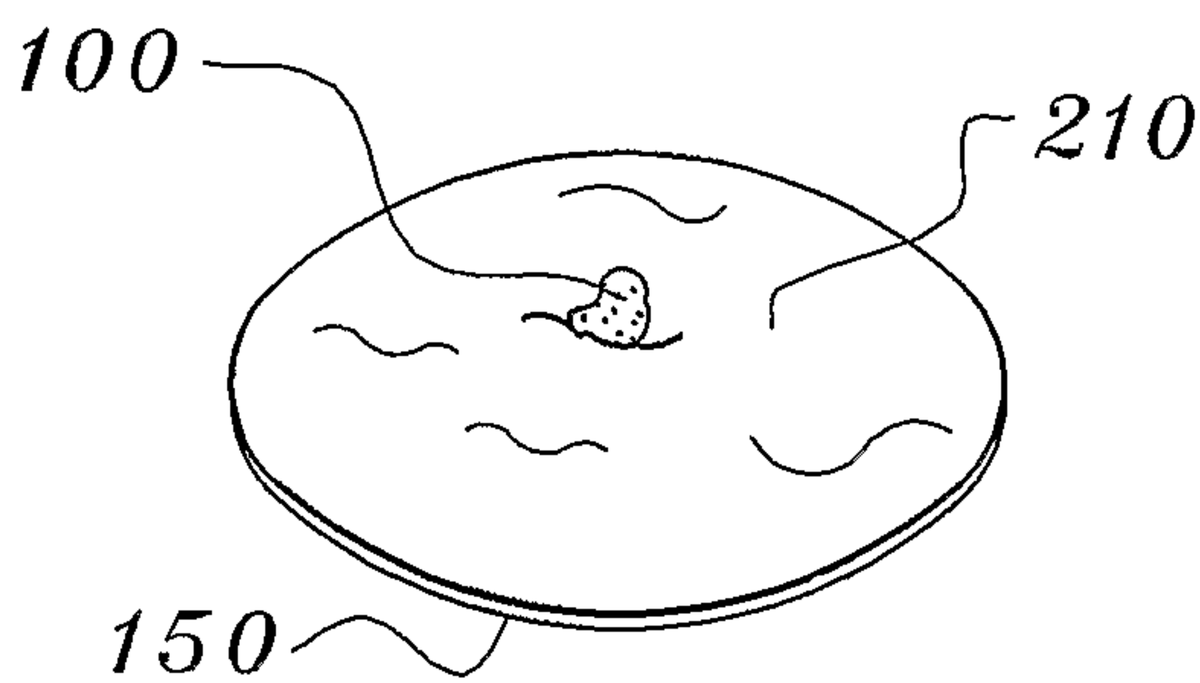


Fig. 5-C

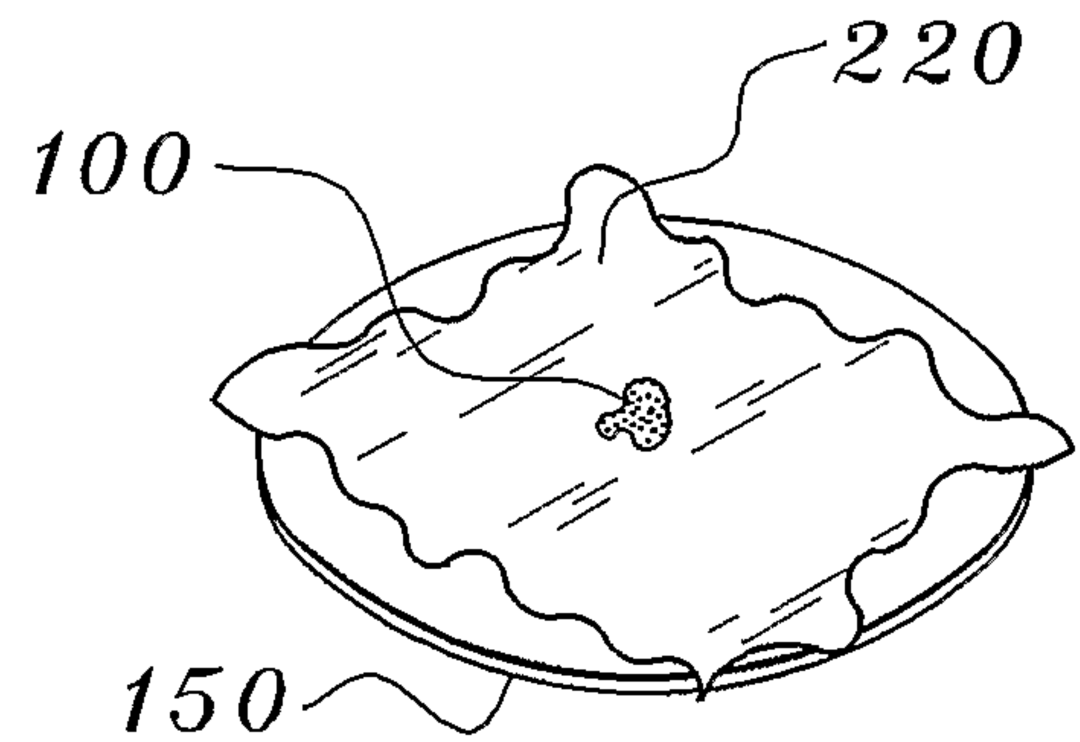


Fig. 5-D

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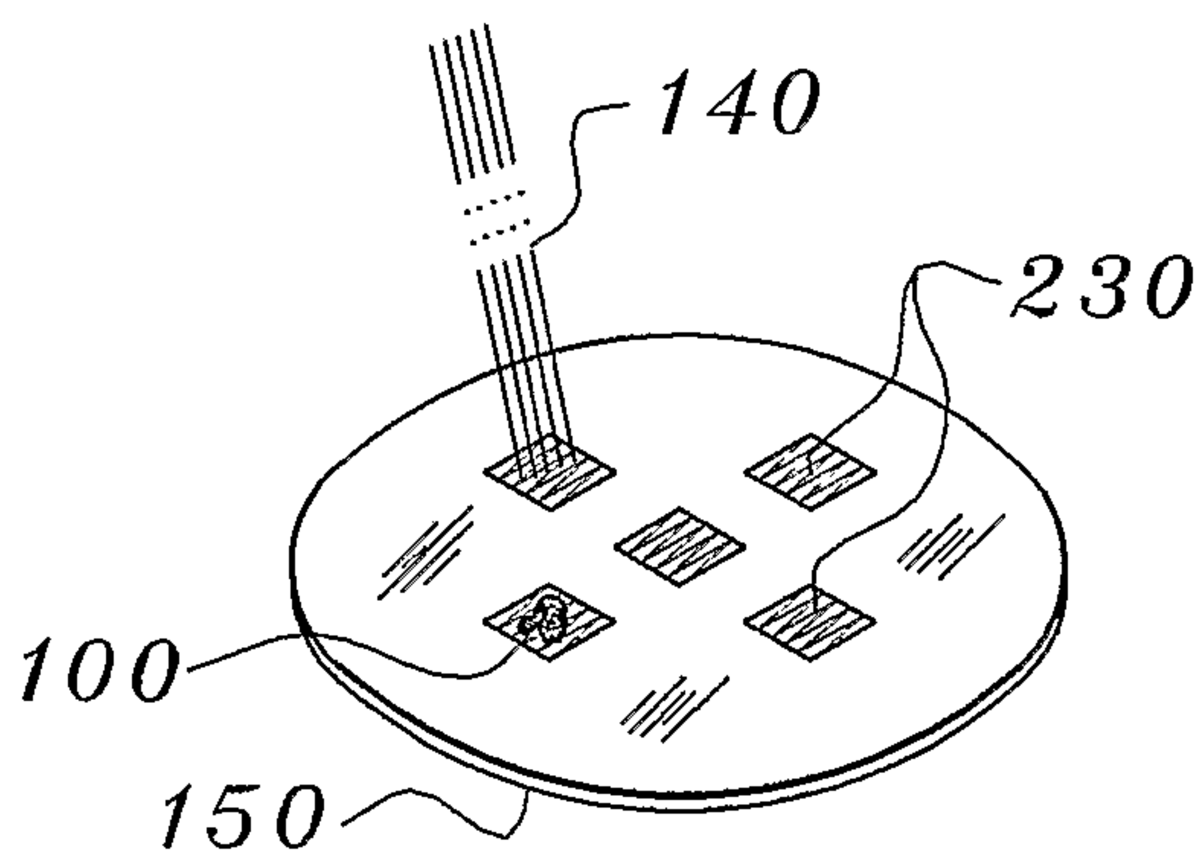


Fig. 5-E

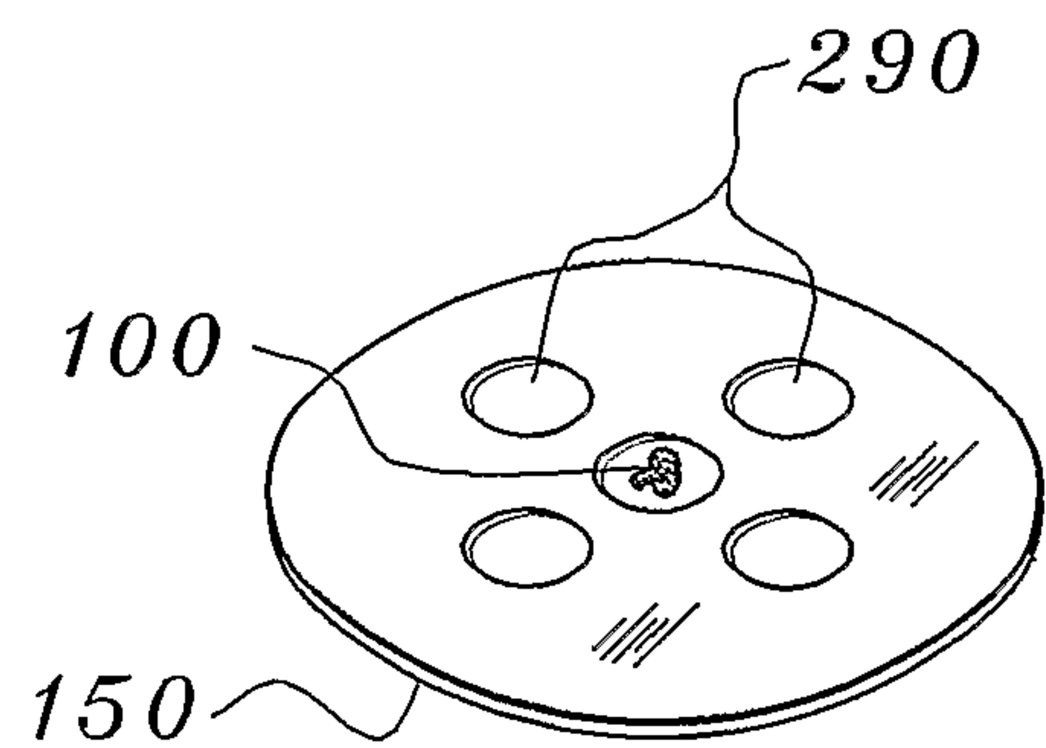


Fig. 5-F

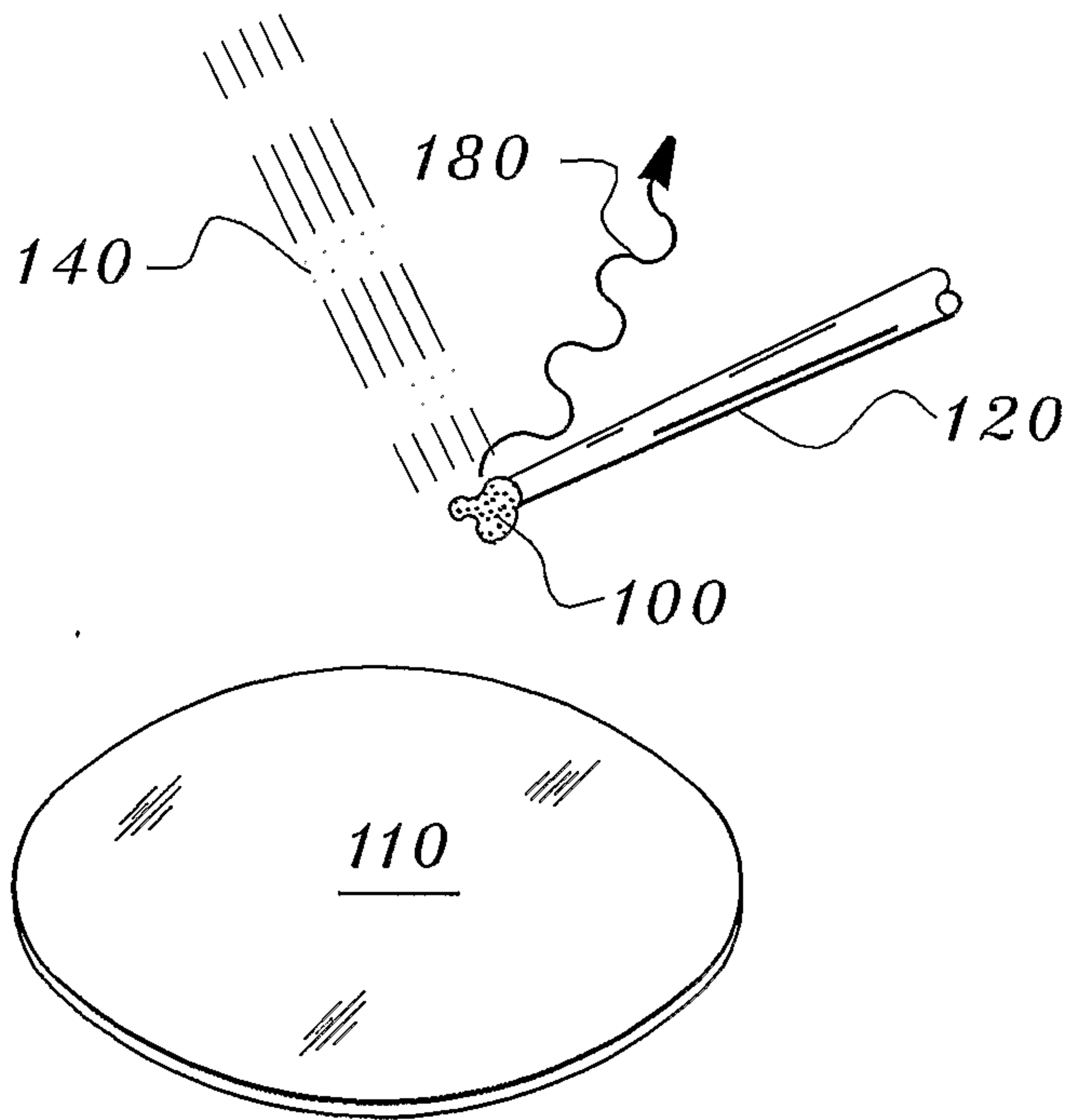


Fig. 6

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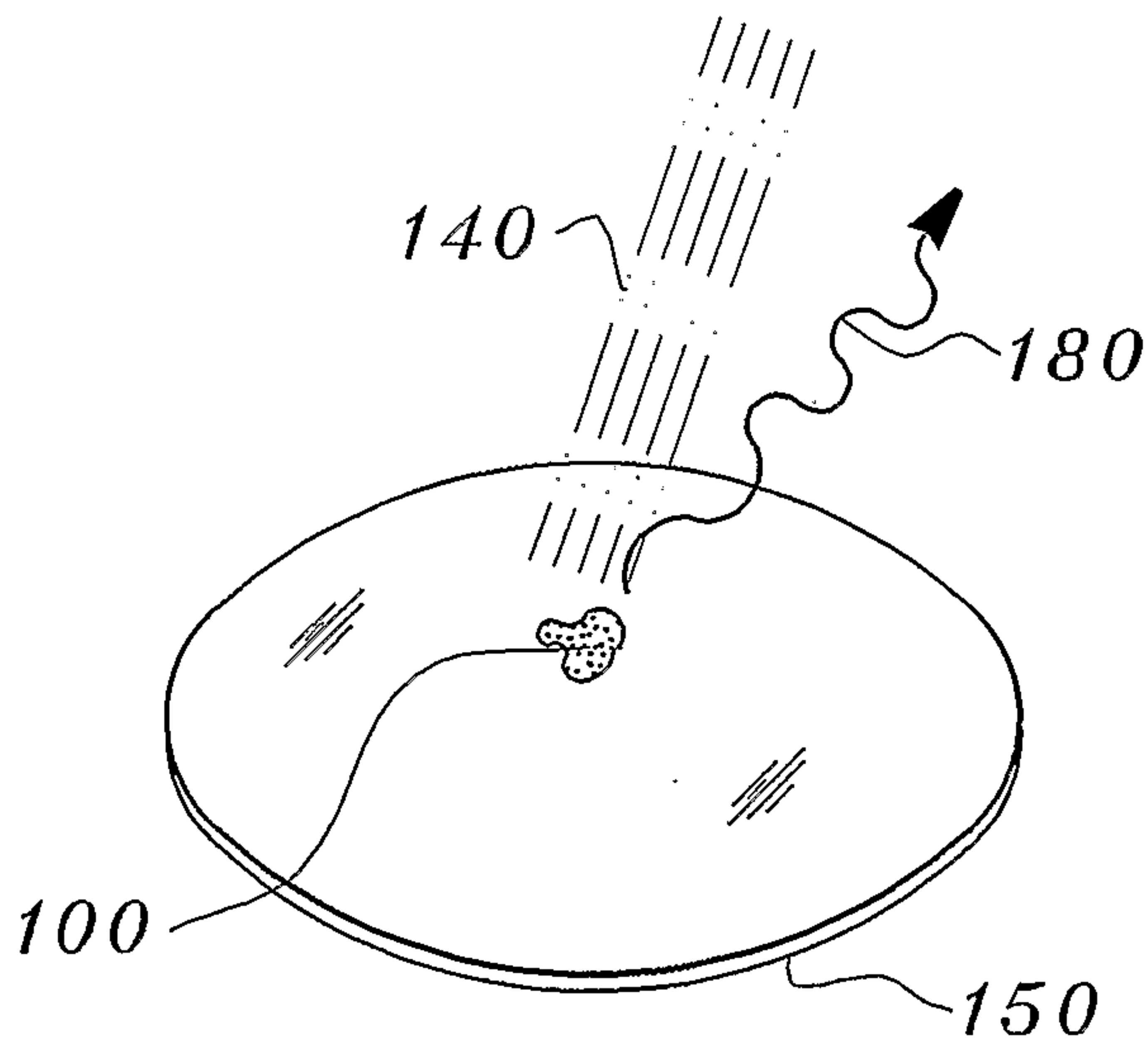
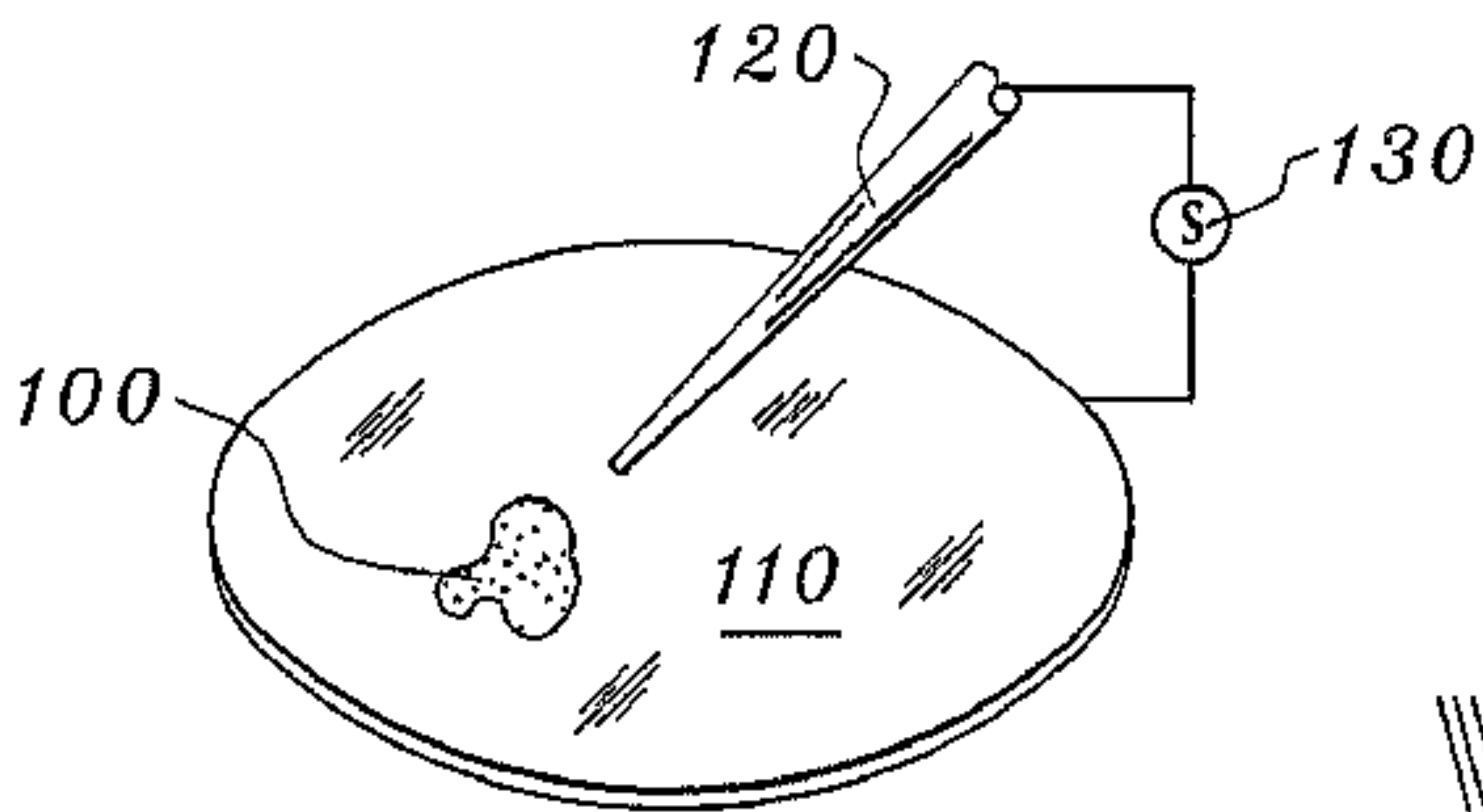
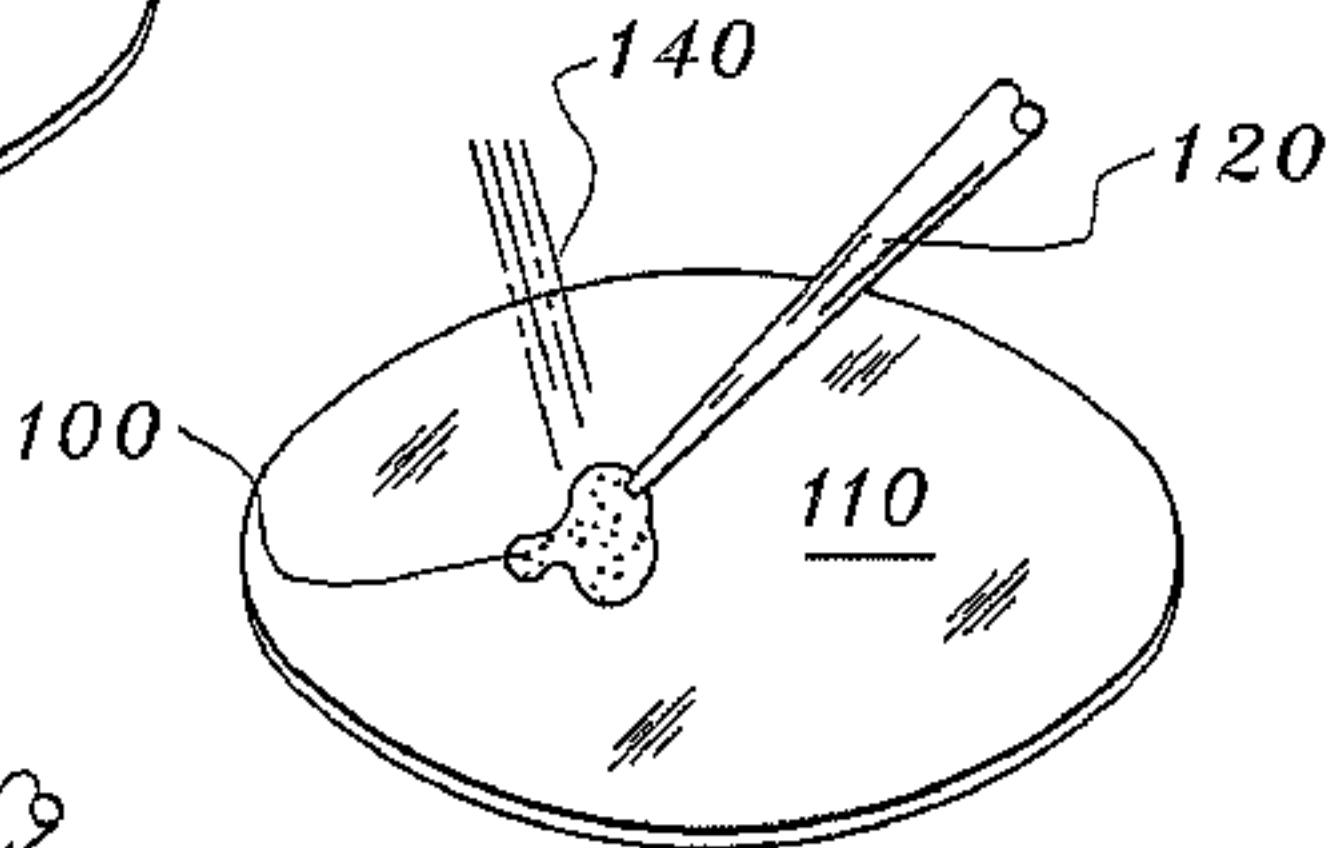



Fig. 7

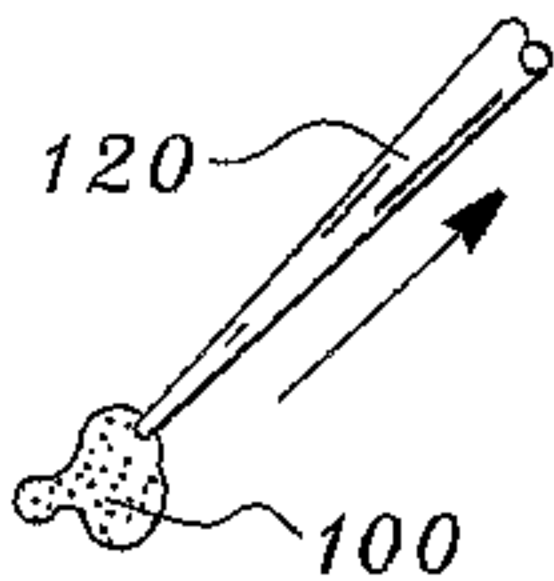
*A*




*B*




*C*




*D*