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**Liu et al.**

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(54) **MICROFABRICATED MICRO FLUID CHANNELS**

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(75) Inventors: **Chang Liu**, Champaign, IL (US); **Kee Ryu**, Urbana, IL (US); **David Bullen**, Urbana, IL (US)

(73) Assignee: **The Board of Trustees of the University of Illinois**, Urbana, IL (US)

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**B01L 3/02** (2006.01)

(52) **U.S. Cl.** ..... **422/100; 422/101; 422/102**

(58) **Field of Classification Search** ..... **422/100-102**  
See application file for complete search history.

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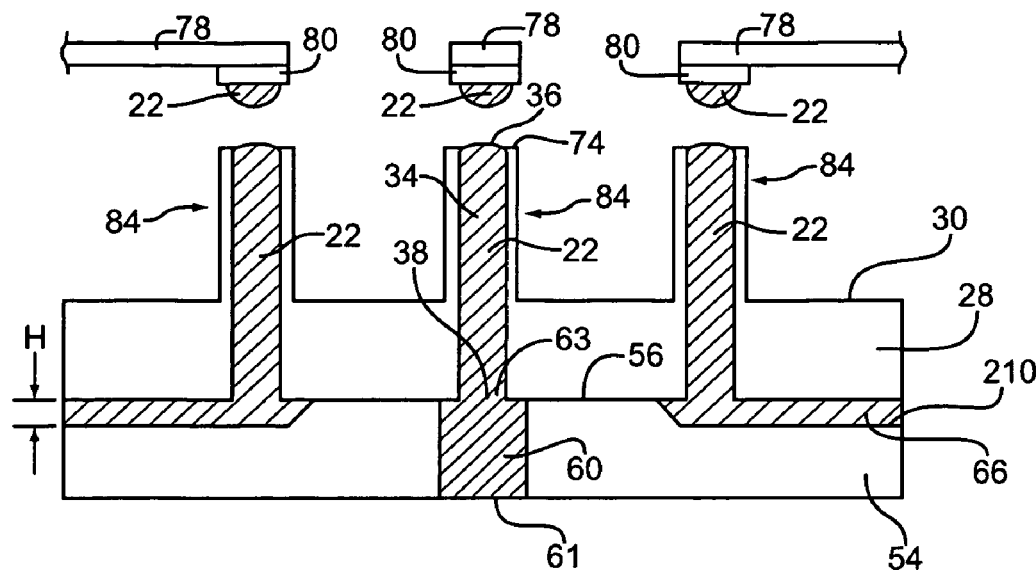
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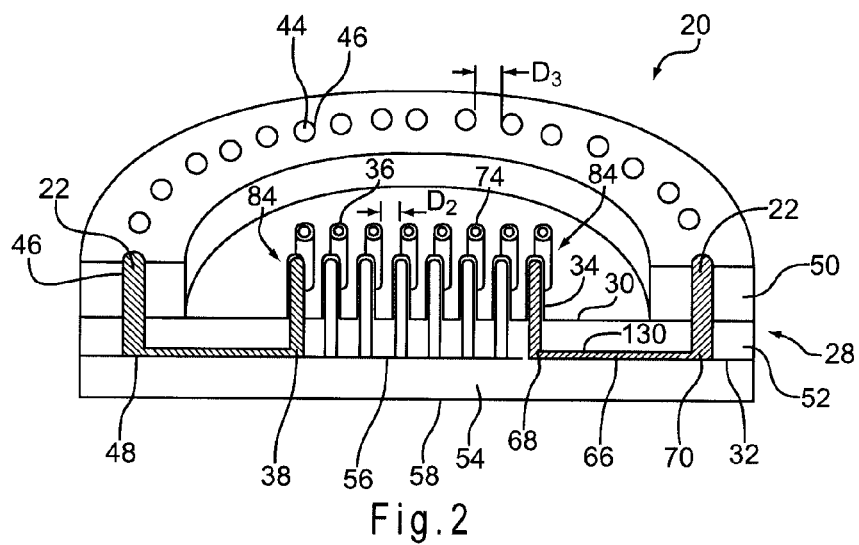
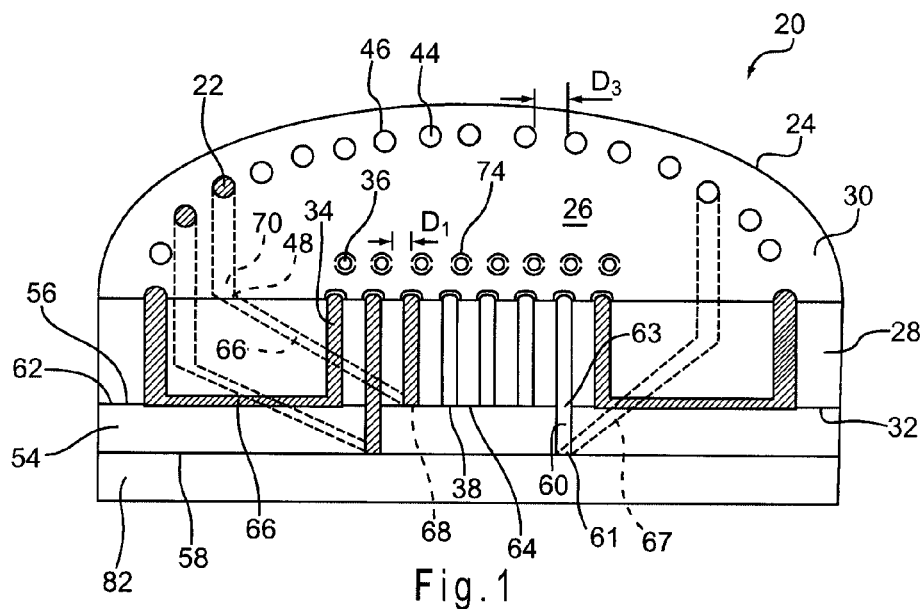
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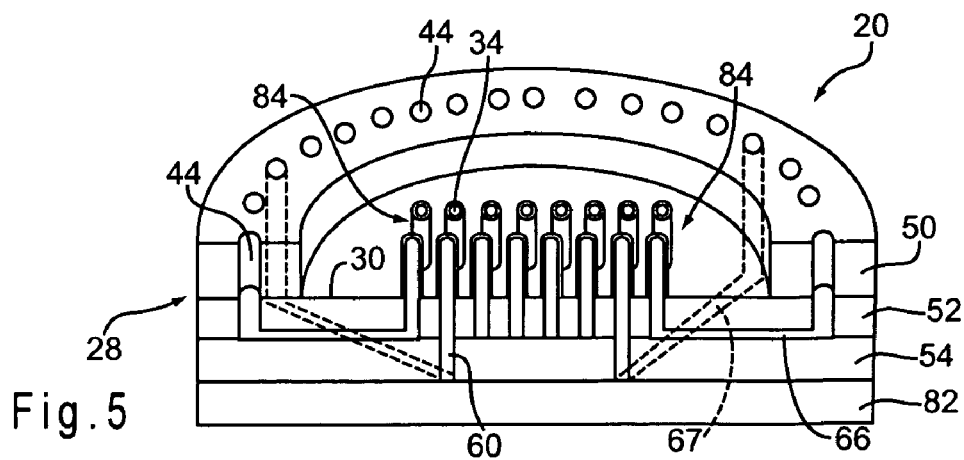
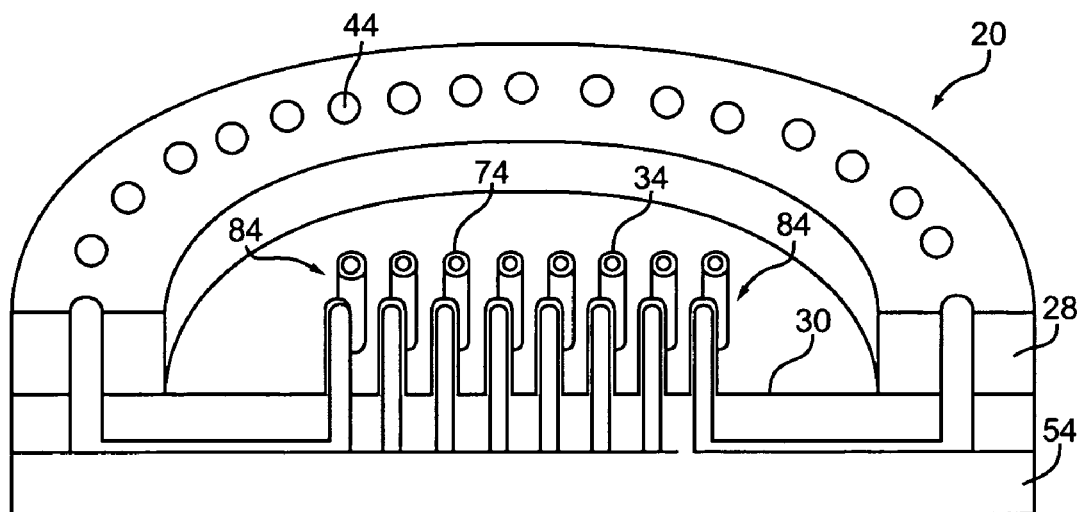
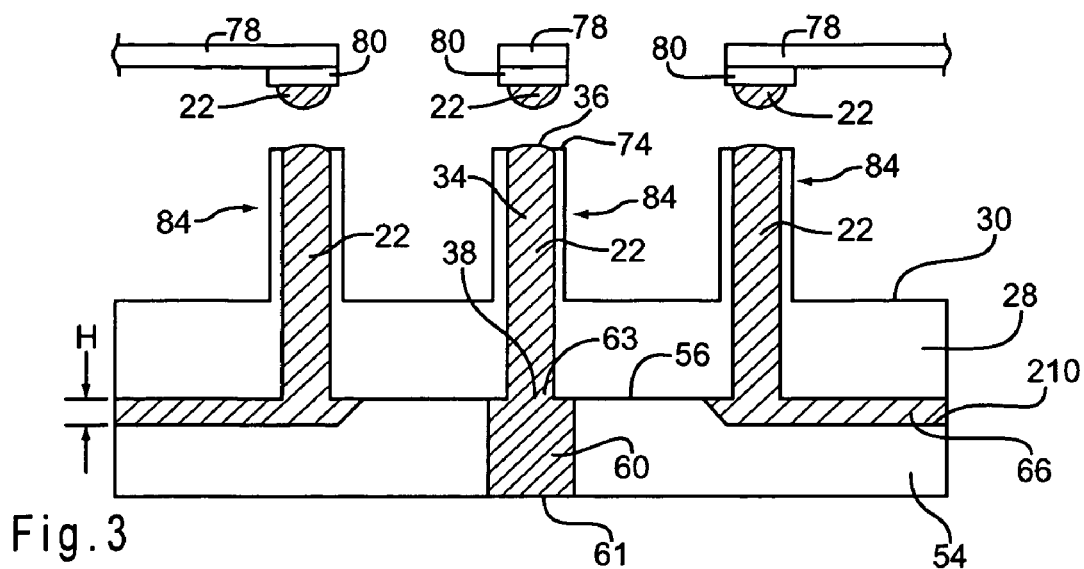
(57) **ABSTRACT**

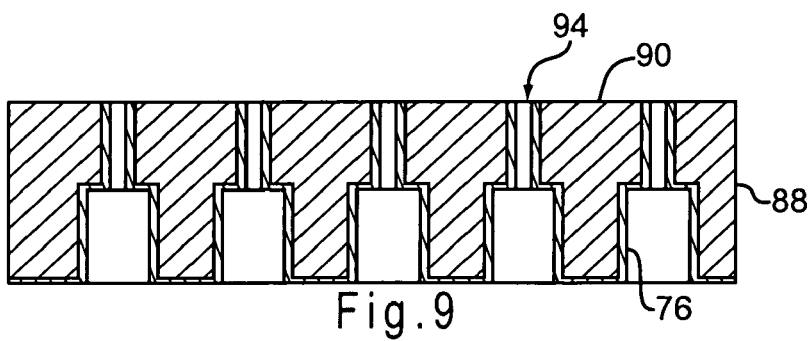
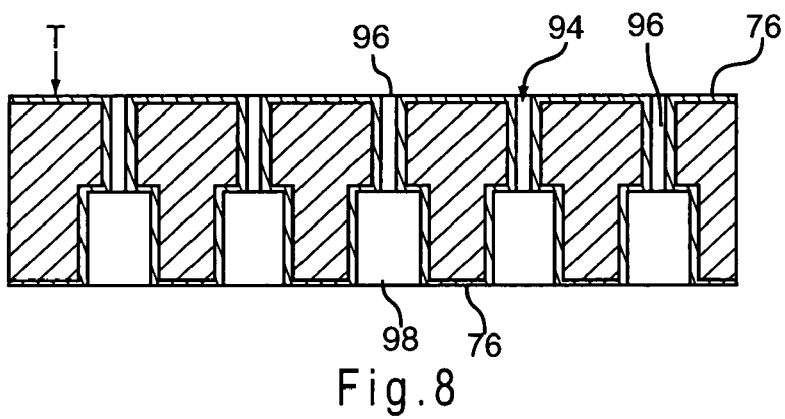
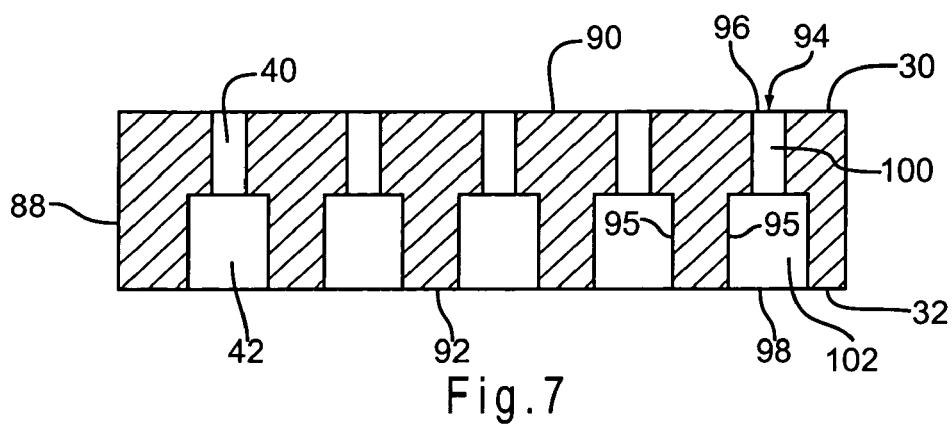
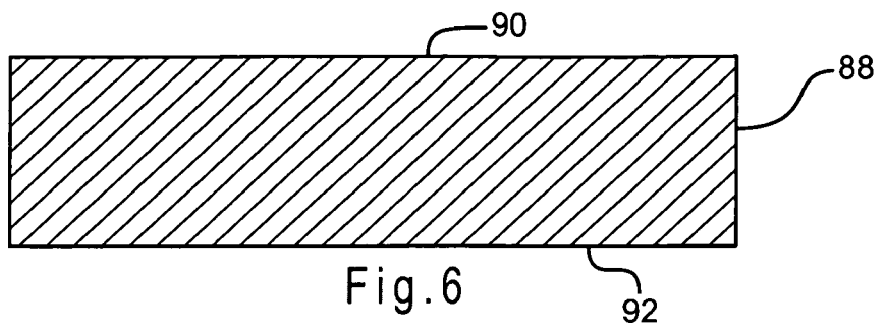
A fluid delivery system including a first substrate having a micro-channel and a well both formed through the first substrate. The fluid delivery system also includes a second substrate and a delivery channel. The second substrate is on the first substrate and the delivery channel is formed between the first and second substrates. The delivery channel provides fluid communication between the micro-channel and the well.

**17 Claims, 4 Drawing Sheets**









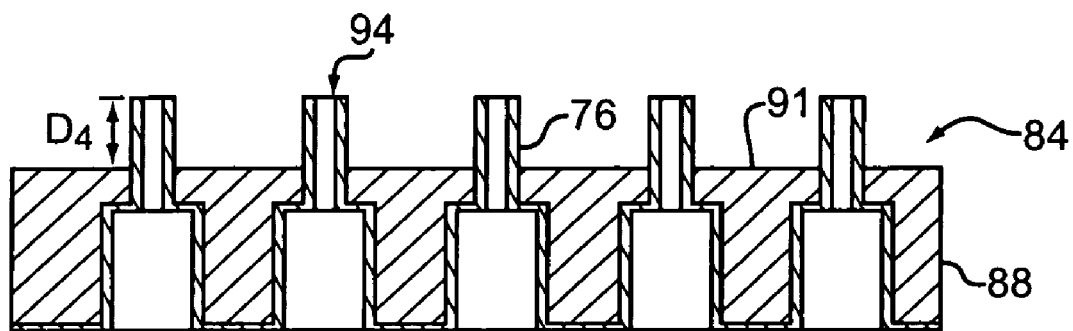


Fig. 10

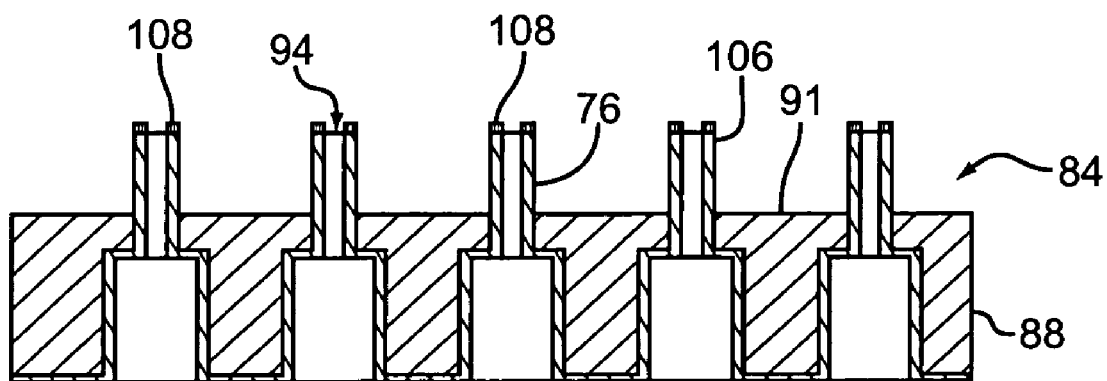


Fig. 11

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## MICROFABRICATED MICRO FLUID CHANNELS

### FEDERALLY SPONSORED RESEARCH OR DEVELOPMENT

This invention was made with Government support under Contract Number DAAD19-00-1-0414 awarded by Army Research Office (ARO), and Contract Number F49620-01-1-0401 awarded by Air Force Office of Scientific Research (AFOSR). The Government may have certain rights in the invention.

### BACKGROUND

This invention relates generally to array chemical transfer devices, and in particular, to a fluid delivery system having an array of micro-channels in fluid connection with an array of wells.

In nanolithography, such as dip pen lithography (DPN) or other types of arrayed chemical transfer methods, it is desirable to provide inks, chemical or biological fluids, to a plurality of probes simultaneously. An arrayed fluid dispensing system with matching spatial configuration to the probe array would allow efficient inking of such an array of probes. Neighboring probes with very small distances between them can receive distinct inks. The probes can then be used to create high density arrays of biochemical substances, such as DNA or protein arrays.

### BRIEF SUMMARY

According to one aspect of the present invention, a method for fabricating a fluid delivery system is provided. The method includes attaching a first substrate and a second substrate to form a delivery channel between the first substrate and the second substrate. The delivery channel provides fluid communication between a first micro-channel and a first well. The first micro-channel and the first well are both in the first substrate.

According to another aspect of the present invention, a fluid delivery system is provided. The fluid delivery system includes a first substrate having a micro-channel and a well both formed through the first substrate. The fluid delivery system also includes a second substrate and a delivery channel. The second substrate is on the first substrate, and the delivery channel is formed between the first and second substrates. The delivery channel provides fluid communication between the micro-channel and the well.

According to another aspect of the present invention, a method for forming a micro-pipette is provided. The method includes etching a top surface of a substrate to remove a portion of the substrate. The substrate has a micro-channel formed through the first substrate, where the top surface is opposed to a bottom surface. The top surface, the bottom surface, and the micro-channel are coated with a support material.

According to another aspect of the present invention, an apparatus for transferring fluid is provided. The apparatus includes a substrate having a top surface opposed to a bottom surface and a layer of support material. The substrate and the layer of support material form a micro-channel. The micro-channel opens at the bottom surface of the substrate and extends above the top surface of the substrate.

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## BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 illustrates a cross-sectional view of a fluid delivery system containing multiple substrates, in accordance with one preferred embodiment of the invention;

FIG. 2 illustrates a cross-sectional view of a fluid delivery system containing multiple substrates, in accordance with one preferred embodiment of the invention;

FIG. 3 illustrates an enlarged cross-sectional view of a portion of a fluid delivery system containing multiple substrates, in accordance with one preferred embodiment of the invention;

FIG. 4 illustrates a cross-sectional view of a fluid delivery system containing multiple substrates, in accordance with one preferred embodiment of the invention;

FIG. 5 illustrates a cross-sectional view of a fluid delivery system containing multiple substrates, in accordance with one preferred embodiment of the invention; and

FIGS. 6–11 illustrate, in cross-section, process steps for the fabrication of a micro-pipette structure for use in a fluid delivery system in accordance with one preferred embodiment of the invention.

It should be appreciated that for simplicity and clarity of illustration, elements shown in the Figures have not necessarily been drawn to scale. For example, the dimensions of some of the elements are exaggerated relative to each other for clarity. Further, where considered appropriate, reference numerals have been repeated among the Figures to indicate corresponding elements.

### DETAILED DESCRIPTION

The present invention includes micro fluid channels. The micro fluid channels are formed by etching partially or completely through a substrate. The micro fluid channels may protrude from the substrate to form micro-pipettes, which may be in the form of an array. The channels may be part of a fluid delivery system, including one or more additional substrates, with channels between the substrates connecting the micro fluid channel to wells having a larger size, allowing for various fluids to easily be delivered to the micro fluid channel. These micro fluid channels may then be used to ink a probe or array of probes, to spot surfaces. This will allow for patterning, with the various fluids, to form, for example high density arrays of biochemical substances.

A “micro fluid channel” or “micro-channel” means a channel having a cross-sectional area of at most 10,000 square microns, more preferably at most 2500 square microns, most preferably at most 100 square microns.

Shown in FIG. 1, in cross-section, is a fluid delivery system 20 which includes a first substrate 28 overlying a second substrate 54. Preferably, the first and second substrate 28, 54 comprise a single crystal silicon substrate; however, first and second substrate 28, 54 may comprise other materials. Preferably, the substrates 28, 54 each have a thickness of 1000 to 200 microns, such as 300 to 500 microns. Preferably, the first and second substrates 28, 54 have top surfaces 30, 56 which are previously processed and cleaned to remove debris and native oxides.

First substrate 28 has a top surface 30 opposed to a bottom surface 32. First substrate 28 also has an outer edge 24 surrounding an inside surface 26, as illustrated in FIG. 1. In one embodiment, the first substrate 28 is formed from a single substrate, as illustrated in FIGS. 1 and 4, while in another embodiment, the first substrate 28 is formed from

multiple substrates, such as an upper substrate **50** and a lower substrate **52**, and then bonded together, as illustrated in FIGS. **2** and **5**.

Upper and lower substrates **50**, **52** may be bonded in one of many ways, such as spin on bonding using photoresist or an adhesive polymer for adhesive bonding, which may be patterned (see for example "VOID-FREE FULL WAFER ADHESIVE BONDING" F. Niklaus, et al.); or high-temperature bonding, for example by heating the substrates together at about 1100° C. Alignment may be achieved using an alignment mark, or using features present on the substrates.

A micro-channel **34** is formed through the first substrate **28** from the top surface **30** to the bottom surface **32**. Preferably, the micro-channel **34** is formed by etching all the way through the first substrate **28** from the top surface **30** to the bottom surface **32**; however other means for forming the micro-channel **34** may be used, such as drilling. Preferably, the micro-channel **34** is formed using anisotropic etching. In one embodiment, the micro-channel **34** is formed by etching through the top surface **30** to form a first channel **40** and then etching through the bottom surface **32** to form a second channel **42**, wherein the first and second channels **40**, **42** are in fluid communication, as illustrated in FIG. **7**. Preferably, the cross-sectional area of the first channel **40** is less than the cross-sectional area of the second channel **42**. More preferably, the ratio of the cross-sectional area of the first channel **40** to the cross-sectional area of the second channel **42** is between 1 to 1 and 1 to 10.

The micro-channel **34** has an inlet **38** for receiving fluid **22** from a delivery channel **66** and an outlet **36** for delivering fluid **22**, preferably, to a probe **78**, as illustrated in FIG. **3**. In one embodiment, the outlet **36** has a cross-sectional area that is generally circular in shape. Preferably, the outlet **36** has a cross sectional area that is at most 10,000 square microns, more preferably at most 2,500 square microns, and most preferably at most 100 square microns. In one embodiment, the outlet **36** has a cross sectional area that is between ten and 10,000 square microns. Preferably, the inlet **38** has a cross-sectional area that is larger than the cross-sectional area of the outlet **36**. The cross-sectional areas and shapes of the inlet **38** and the outlet **36** may be freely chosen, using patterning techniques, such as those used to pattern semiconductor substrates.

Preferably, the first substrate **28** includes a hydrophobic surface **74** surrounding the outlet **36**, as illustrated in FIG. **1**. The hydrophobic surface **74** prevents fluid **22** from overflowing and unintentionally exiting the outlet **36**, and possibly causing cross-contamination between fluids **22**. The hydrophobic surface **74** can be formed in one of many ways, such as coating the outlet **36** of the micro-channel **34** with a hydrophobic material, for example a silane or thiol, such as 1-octadecanethiol (ODT), or a photoresist. Preferably, an array of micro-channels **34** is formed through the first substrate **28** from the top surface **30** to the bottom surface **32**, as illustrated in FIG. **1**. The distance  $D_1$  between adjacent micro-channels **34** is preferably at most 1000 microns, more preferably at most 600 microns, most preferably at most 200 microns.

A well **44** is formed through the first substrate **28** from the top surface **30** to the bottom surface **32**. Preferably, the well **44** is formed by etching all the way through the first substrate **28** from the top surface **30** to the bottom surface **32**; however other means for forming the well **44** may be used, such as drilling. Preferably, the well **44** is formed using reactive ion etching. The well **44** has an inlet **46** for receiving fluid **22** from a fluid delivery device, such as a

conventional pipette or pump. The well **44** also includes a well outlet **48** for delivering fluid **22** to the delivery channel **66**, as illustrated in FIG. **1**. In one embodiment, the well inlet **46** has a cross-sectional area that is generally circular in shape. Preferably, the well inlet **46** has a cross-sectional area that is greater than the cross-sectional area of the micro-channel outlet **36**. Forming the well inlet **46** with a cross-sectional area that is larger than the cross-sectional area of the micro-channel outlet **36** allows for fluids **22** to be easily injected into the well inlet **46**, and yet still be deliverable, through the smaller micro-channel outlet **36**, to a probe **78**. Preferably, the well inlet **46** has a cross sectional area that is between 1 and 20 square millimeters, more preferably between 3 and 12 square millimeters, and most preferably between 5 and 10 square millimeters. In one embodiment, the distance  $D_3$  between adjacent wells **44** is greater than the distance  $D_1$  between adjacent micro-channels **34** or the distance  $D_2$  between adjacent micro-pipettes **84**, as illustrated in FIGS. **1** and **2**.

Preferably, the array of wells **44** is formed through the first substrate **28** from the top surface **30** to the bottom surface **32**, as illustrated in FIG. **1**. Preferably, the array of wells **44** is formed near the outer edge **24** of the first substrate **28**, while the array of micro-channels **34** is formed at the inside surface **26** of the first substrate **28**, as illustrated in FIG. **1**.

Second substrate **54** has a top surface **56** opposed to a bottom surface **58**. Second substrate **54** also has an outer edge **62** surrounding an inside surface **64**, as illustrated in FIG. **1**. Second substrate **54** is positioned so that the top surface **56** is on the bottom surface **32** of the first substrate **28**. In one embodiment, an extended channel **60** is formed through the second substrate **54** from the top surface **56** to the bottom surface **58**. Preferably, the extended channel **60** is formed by etching all the way through the second substrate **54** from the top surface **56** to the bottom surface **58**; however, other means for forming the extended channel **60** may be used, such as drilling. Preferably, the extended channel **60** is formed using reactive ion etching. The extended channel **60** has an inlet **61** for receiving fluid **22** from a secondary delivery channel **67** and an outlet **63** for delivering fluid **22** to the inlet **38** of the micro-channel **34**, as illustrated in FIGS. **1** and **3**. The secondary delivery channel **67** is formed between the second substrate **54** and a third substrate **82**, as illustrated in FIG. **1**. In one embodiment, the outlet **63** has a cross-sectional area that is generally circular in shape.

The delivery channel **66** is formed between the first and second substrates **28**, **54**, as illustrated in FIGS. **1** and **2**. Preferably, the height  $H$  of the delivery channel, as illustrated in FIG. **3**, is between one and twenty microns, and more preferably, at most ten microns. The delivery channel **66** may be formed in one of many ways. In one embodiment, the delivery channel **66** includes a groove **210** that is formed on the top surface **56** of the second substrate **54**. In one embodiment, the delivery channel **66** includes a groove **130** that is formed on the bottom surface **32** of the first substrate **28**. In one embodiment, the delivery channel **66** includes a first groove that is formed on the top surface **56** of the second substrate **54** and a second groove that is formed on the bottom surface **32** of the first substrate **28**. The first substrate **28** is aligned with the second substrate **54** so that the delivery channel **66** allows for fluid to travel between the micro-channel **34** and the well **44**. The delivery channel **66** includes an inlet **70** for receiving fluid and an outlet **68** for delivering fluid **22**. The delivery channel inlet **70** receives fluid **22** from the well outlet **48** of the well **44**, while the

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delivery channel outlet 68 delivers fluid 22 to the inlet 38 of the micro-channel 34. The delivery channel inlet 70 is adjacent to the well outlet 48, and the delivery channel outlet 68 is adjacent to the inlet 38, as illustrated in FIG. 1.

In one embodiment, the micro-channel 34 extends both above and below the top surface 30 of the first substrate 28, as illustrated in FIGS. 2-5. In this embodiment, the micro-channel 34 may be formed entirely from the first substrate 28, a portion of the micro-channel 34 may be formed from the first substrate 28, or the micro-channel 34 may be formed from a second material. The second material may be any material that can be formed or coated onto a surface, such as those used in semiconductor processing. Examples include oxides, such as silicon oxide and silicon oxynitride, nitrides such as silicon nitride and titanium nitride, metals such as tungsten and gold, and polymers. Preferably, the micro-channel 34 is formed from the second material, wherein the second material extends both above and below the top surface 30 of the first substrate 28.

In one embodiment, a method for fabricating the fluid delivery system 20 is disclosed. Referring to FIG. 1 the fluid delivery system 20 is fabricated by attaching the first substrate 28 and the second substrate 54 to form the delivery channel 66, wherein the delivery channel 66 provides fluid communication between the micro-channel 34 and the well 44. Preferably, the first substrate 28 is aligned with the second substrate 54 so that the inlet 70 of the delivery channel 66 is adjacent the outlet 48 of the well 44, while the outlet 68 of the delivery channel 66 is adjacent the inlet 38 of the micro-channel 34. In this way, the micro-channel 34 is in fluid connection with the well 44, and thus fluid 22 may travel from the inlet 46 of the well 44 through the well 44, the delivery channel 66, and the micro-channel 34, only to exit at the outlet 36 of the micro-channel 34. Preferably, either alignment marks or existing features on one or both of the first and second substrates 28, 54 are used to align the first substrate 28 with the second substrate 54.

Once the delivery channel 66 is formed, the fluid 22 is delivered to the well 44. The fluid 22 travels down the well 44, through the delivery channel 66, and up the micro-channel 34 to the outlet 36. Fluid 22 can be forced up into the micro-channel 34 by pumping the fluid 22 through the well 44 and the delivery channel 66. Fluid 22 may be pumped through the well 44 and the delivery channel 66 using a variety of techniques, such as by creating a pressure differential between the inlet 46 and the outlet 36, or simply by capillary action. Preferably, the fluid 22 is kept in the micro-channel 34 and prevented from unintentionally exiting the micro-channel 34 by creating a hydrophobic surface 74 adjacent the outlet 36 of the micro-channel 34. Once the micro-channel 34 is filled with fluid 22, the fluid 22 can then be transferred to a probe 78, such as an SPM (Scanning Probe Microscopy) probe, and more specifically to the tip 80 of the probe 78, as illustrated in FIG. 3.

In one embodiment, a micro-pipette 84 forms the micro-channel 34. Preferably, a portion of the micro-pipette 84 extends both above and below the top surface 30 of the first substrate 28, as illustrated in FIG. 2. Preferably, the fluid delivery system 20 includes an array of micro-pipettes 84, as illustrated in FIGS. 2-5, allowing for multiple fluids 22 to be dispensed from the array of micro-channels 34.

In one embodiment, a method for fabricating the micro-pipette 84 is disclosed, as illustrated in FIGS. 6-11. Referring to FIG. 6, a substrate 88, such as first substrate 28, is provided. Preferably, the substrate 88 has a top surface 90 which is previously processed and cleaned to remove debris and native oxides. The top surface 90 is opposed to a bottom

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surface 92. Preferably, the bottom surface 92 is also previously processed and cleaned to remove debris and native oxides.

Referring to FIG. 7, a channel 94, such as a micro-channel 34, having a wall 95 is formed through the substrate 88 from the top surface 90 to the bottom surface 92. Preferably, the channel 94 is formed by etching all the way through the substrate 88 from the top surface 90 to the bottom surface 92; however, other means for forming the channel 94 may be used, such as drilling. Preferably, the channel 94 is formed using reactive ion etching. In one embodiment, the channel 94 is formed by etching through the top surface 92 to form a first channel 100 and then etching through the bottom surface 92 to form a second channel 102, wherein the first and second channels 100, 102 connect, as illustrated in FIG. 7. The channel 94 has an inlet 98 for receiving fluid, such as fluid 22, and an outlet 96 for delivering fluid to, for example, a probe, such as probe 78. In one embodiment, the outlet 96 has a cross-sectional area that is generally circular in shape. In yet another embodiment, the inlet 98 has a cross-sectional area that is larger than the cross-sectional area of the outlet 96. The cross-sectional areas and shapes of the inlet 98 and the outlet 96 may be freely chosen, using patterning techniques, such as those used to pattern semiconductor substrates.

After forming the channel 94, the top surface 90, the bottom surface 92, and the wall 95 of the channel 94 are all coated with a layer 76 of support material, as illustrated in FIG. 8. The support material may include any material that can be formed or coated onto a surface, such as those used in semiconductor processing. Examples include oxides, such as silicon oxide and silicon oxynitride, nitrides such as silicon nitride and titanium nitride, metals such as tungsten and gold, and polymers. The support material may be formed by chemical reaction with the substrate 88, for example by oxidation, or by coating, for example with chemical vapor deposition or oblique angle physical vapor deposition. Preferably, the support material is different from the material contained in the substrate 88. Multiple materials may also be used, and these may be applied with the first support material, or they may be applied after further processing steps. In one embodiment, the layer 76 is silicon dioxide which is formed by reacting the substrate 90, preferably made of silicon, with oxygen.

After forming the layer 76, a portion of the layer 76 is removed. Preferably, the layer 76 is removed from the top surface 90 to expose the top surface 90 of the substrate 88. In one embodiment, the layer 76 is removed from the bottom surface 92 to expose the bottom surface 92 of the substrate 88. Layer 76 may be removed in one of a number of ways, such as the use of chemical-mechanical polishing or etching. The channel 94 may be filled with a protectant, such as wax, to avoid damaging the channel 94 with a polishing agent. The protectant may be removed with a solvent, such as acetone.

Upon removing the layer 76, the substrate 88 is then etched to remove a portion of either the top surface 90 or the bottom surface 92 of the substrate 88, as illustrated in FIG. 10. Preferably, the substrate 88 is etched to a depth  $D_4$ , as illustrated in FIG. 10, of between 50 and 500 microns, and more preferably, a depth of at most 150 microns. Various types of etching may be used to remove a portion of the substrate 88, such as wet etching with ethylene diamine pyrocatechol or potassium hydroxide, or dry etching. Preferably, the top surface 90 is etched to remove a portion of the substrate 88 and create an etched surface 91 which is closer to the bottom surface 92 than the original top surface 90.



Preferably, upon etching the substrate **88**, a hydrophobic material is applied to the surface **106** surrounding the outlet **96** to create a hydrophobic surface **108**, as illustrated in FIG. **11**.

The individual processing steps used in accordance with the present invention are well known to those of ordinary skill in the art, and are also described in numerous publications and treatises, including: *Encyclopedia of Chemical Technology, Volume 14* (Kirk-Othmer, 1995, pp. 677-709); *Semiconductor Device Fundamentals* by Robert F. Pierret (Addison-Wesley, 1996); *Silicon Processing for the VLSI Era* by Wolf (Lattice Press, 1986, 1990, 1995, vols 1-3, respectively); and *Microchip Fabrication: A Practical Guide to Semiconductor Processing* by Peter Van Zant (4<sup>th</sup> Edition, McGraw-Hill, 2000). In order to etch through the substrate, techniques such as deep ion etching may be used (also known as the Bosch process).

The fluid delivery system may be used to form patterns using fluids on surfaces. For example, if the fluid is an ink, then a surface, such as paper, could be printed with the probes after they have received the ink, to form microprinting. Biological arrays may similarly be formed, for example by using fluids containing biological compounds, such as nucleotides (RNA, DNA, or PNA), proteins (enzymes, antibodies, etc.), lipids, carbohydrates, etc. to spot a substrate, such as glass, silicon, or polymers. Since each micro-channel may be supplied for a dedicated well, very complex arrays may be produced quickly.

Numerous additional variations in the presently preferred embodiments illustrated herein will be determined by one of ordinary skill in the art, and remain within the scope of the appended claims and their equivalents. For example, while the examples provided above relate to silicon-based semiconductor substrates, it is contemplated that alternative semiconductor materials can likewise be employed in accordance with the present invention, and that the semiconductor substrates may be undoped, P-doped, or N-doped. Suitable materials for the substrates include but are not limited to silicon, gallium arsenide, germanium, gallium nitride, aluminum phosphide,  $\text{Si}_{1-x}\text{Ge}_x$  and  $\text{Al}_x\text{Ga}_{1-x}\text{As}$  alloys, wherein  $x$  is greater than or equal to zero and less than or equal to one, the like, and combinations thereof. Additional examples of materials for use in accordance with the present invention are set forth in *Semiconductor Device Fundamentals* by Robert F. Pierret (p. 4, Table 1.1, Addison-Wesley, 1996).

Although the invention has been described and illustrated with reference to specific illustrative embodiments thereof, it is not intended that the invention be limited to those illustrative embodiments. Those skilled in the art will recognize that variations and modifications can be made without departing from the spirit of the invention.

The invention claimed is:

1. A fluid delivery system, comprising:

a first substrate comprising:

a first micro-channel comprising a first micro-channel outlet,

a first well,

a second micro-channel comprising a second micro-channel outlet, and

a second well, each formed through the first substrate;

a second substrate on the first substrate;

a first delivery channel formed between the first and the second substrates, the first delivery channel establishing fluid communication between the first micro-channel and the first well; and

a second delivery channel establishing fluid communication between the second micro-channel and the second well; and

a scanning probe microscopy probe for receiving fluid from the micro-channels, wherein the probe is moveable to and from the micro-channel outlets,

where the first and second micro-channel outlets are separated by at most 1000 microns.

2. The system of claim 1, wherein at least one of the delivery channels comprises a groove formed on a surface of the second substrate.

3. The system of claim 1,

where each of the wells has an inlet for receiving fluid, each of the micro-channels has an outlet for delivering fluid, and

a cross-sectional area of at least one of the well inlets is greater than a cross sectional area of at least one of the micro-channel outlets.

4. The system of claim 3, wherein the cross sectional area of each of the well inlets is between one and twenty square millimeters, and the cross sectional area of each of the micro-channel outlets is between ten and 10,000 square microns.

5. The system of claim 3, further comprising a hydrophobic surface surrounding each of the micro-channel outlets.

6. The system of claim 3, wherein each of the micro-channel extends above and below a surface of the first substrate.

7. The system of claim 3, wherein the first substrate comprises a plurality of substrates.

8. The system of claim 1, further comprising a third substrate on the second substrate.

9. The system of claim 8, the second substrate further comprising an extended channel establishing fluid communication between the second micro-channel and the second delivery channel, the extended channel formed through the second substrate; and

the second delivery channel formed between the second and the third substrates.

10. The system of claim 1, where at least one delivery channel comprises a groove formed on a surface of the first substrate.

11. The system of claim 1, where the first substrate has a thickness of at most 500 microns.

12. The system of claim 1, where the first substrate is bonded to the second substrate by spin-on bonding or high temperature bonding.

13. The system of claim 1, where the first and second micro-channels form a micro-pipette.

14. The system of claim 1, where the micro-channels are formed by reactive ion etching.

15. The system of claim 14, where the micro-channels are formed by a first etch on a top surface of the first substrate and a second etch on a bottom surface of the first substrate.

16. The system of claim 15, where the first etch forms a first micro-channel width and the second etch forms a second micro-channel width, the first micro-channel width in fluid communication with the second micro-channel width.

17. The system of claim 1, where the distance between the first and second wells is greater than the distance between the first and second micro-channels.