

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
15 November 2007 (15.11.2007)

PCT

(10) International Publication Number
WO 2007/128045 A1

(51) International Patent Classification:

B81B 7/02 (2006.01) **C30B 30/06** (2006.01)
B06B 1/06 (2006.01) **G01N 1/28** (2006.01)
C30B 29/34 (2006.01) **H01L 41/09** (2006.01)

(21) International Application Number:

PCT/AU2007/000575

(22) International Filing Date: 2 May 2007 (02.05.2007)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:

2006902259 2 May 2006 (02.05.2006) AU

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(81) Designated States (unless otherwise indicated, for every

kind of national protection available): AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BH, BR, BW, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IS, JP, KE, KG, KM, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PG, PH, PL, PT, RO, RS, RU, SC, SD, SE, SG, SK, SL, SM, SV, SY, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

(84) Designated States (unless otherwise indicated, for every

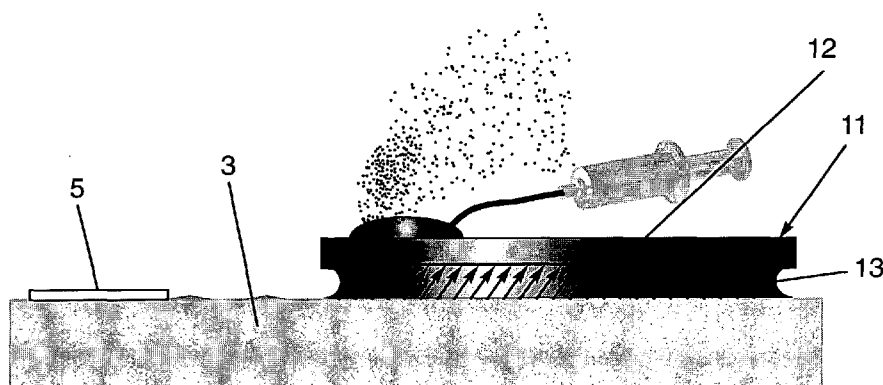
kind of regional protection available): ARIPO (BW, GH, GM, KE, LS, MW, MZ, NA, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IS, IT, LT, LU, LV, MC, MT, NL, PL, PT, RO, SE, SI, SK, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

Published:

— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: MICROFLUIDIC SYSTEMS USING SURFACE ACOUSTIC ENERGY AND METHOD OF USE THEREOF



(57) Abstract: A microfluidic system including a piezoelectric substrate (1), a wave generation means (5) for generating a wave in the piezoelectric substrate, and a rigid secondary substrate (11) coupled to said piezoelectric substrate (1) and providing a working surface (12) for the system, the secondary substrate (11) being formed of a relatively chemically inert material, and the coupling of the piezoelectric substrate (1) with the secondary substrate (12) being provided by a fluid coupling layer (13) located between the piezoelectric substrate (1) and the secondary substrate (12) overlaying the piezoelectric substrate (1). In one application, the system may be used to synthesize zeolite nanocrystals via atomization of a liquid droplet (15).

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**MICROFLUIDIC SYSTEMS USING SURFACE ACOUSTIC ENERGY AND
METHOD OF USE THEREOF**

The present invention generally relates to microfluidic systems and methods of using such systems, and is in particular directed to microfluidic systems using acoustic energy for the manipulation of fluid and particles suspended within that fluid.

The ongoing development and research in microfluidic processes and systems is driven by the many benefits that can be achieved by conducting processes on fluids at a microscale. In particular, in fluid analysis applications, only small volumes of fluid are required. Also, only small quantities of the associated reagents and analytes are required leading to cost savings. In addition, there are lower power requirements to operating such systems. Furthermore, the potential of mass production of such systems using integrated circuit fabrication methods make them attractive as relatively low cost, portable and disposable diagnostic devices. Greater control can be achieved in the mixing of fluids as the mixing is primarily due to diffusion between the fluids. These systems can also be used in biomedical applications where biological material such as erythrocytes or bacteria, need to be concentrated within the fluid with little to no damage of that material. Many other existing and potential applications are also envisaged for such microfluidic systems.

A simple microfluidic system would typically include a substrate formed of material such as silicon, glass, polymeric film or thermoplastic in which is etched, laser cut or moulded microfluidic channels. Such channels would typically have at least one dimension of less than 1 mm. A cover may also be provided over the channels to enclose them. A syringe or a microelectromechanical system (MEMS) may then be used to transport fluids, and any associated reagents or analytes through these channels.

More sophisticated microfluidic systems utilise piezoelectric actuators which are actuated for vibration using electrical excitation. In one such system, capillaries are mounted on or mechanically coupled to such an actuator to thereby allow for ultrasonic vibration of the capillary. This has a particular application in the concentration of biological material within pressure nodes located within the acoustic standing wave generated by the vibration of the

capillary. The major disadvantages of such capillary based systems are difficulties in pumping fluids through the capillaries to reliably perform what are routine laboratory functions on the micro-scale and the subsequent removal of the fluid and the concentrated material from the capillaries.

5 All the above described applications use a continuous flow of fluid through channels or capillaries. It is also possible to conduct microfluidic processes on individual droplets of fluid. These droplets may be applied directly to the surface of a piezoelectric substrate and may then be directly exposed to vibrations generated within the substrate using radio frequency (RF) pulsed excitation. One
10 such system uses a piezoelectric substrate upon which the surface of the piezoelectric substrate is located at least one interdigital electrode. Application of a RF input to the electrodes generates within the piezoelectric substrate surface a surface acoustic wave (SAW), also known as a "Rayleigh" wave. The SAW excitation of the substrate surface acts to displace or manipulate one or more
15 liquid droplets located on that surface.

Lithium Niobate (LiNbO_3) is generally used in such applications because of its good performance in SAW generation. This material is however susceptible to damage from contact to fluids that are corrosive in nature. Such fluids therefore cannot be placed in direct contact with the substrate. Furthermore, it is not
20 practical to produce channels within such a substrate limiting its applicability in continuous flow microfluidic applications.

It would therefore be advantageous to be able to provide a microfluidic system and a method of using such systems which overcomes at least one of the above-noted disadvantages.

25 Any discussion of documents, systems, acts or knowledge in this specification is included to explain the context of the invention. It should not be taken as an admission that any of the material formed part of the prior art base or the common general knowledge in the relevant art in or any other country on or before the priority date of the claims herein.

30 With this in mind, according to one aspect of the present invention there is provided a microfluidic system including:

a piezoelectric substrate;

a wave generation means for generating a wave in the piezoelectric substrate; and

a rigid secondary substrate coupled to said piezoelectric substrate and providing a working surface for the system, the secondary substrate being formed
5 of a relatively easily fabricated and/or chemically inert material;

the coupling of the piezoelectric substrate with the secondary substrate being provided by a fluid coupling layer located between the piezoelectric substrate and the secondary substrate overlaying the piezoelectric substrate.

The wave generation means may generate a surface acoustic wave (SAW)
10 in the surface of the piezoelectric substrate. This wave may then be transferred to the surface of the rigid secondary substrate. It is however also envisaged that other forms of waves could be used in this system to deliver acoustic energy to the secondary substrate including bulk acoustic waves (BAW), surface-skimming bulk waves (SSBW) and shear surface acoustic waves (SH-SAW).

15 The rigid substrate may be made from glass, SiO₂, thermoplastic or other rigid materials. These materials are relatively chemically inert so as not to react to contact with corrosive fluids. The use of a chemically inert rigid substrate allows the microfluidic system to be used for corrosive fluids.

Furthermore, these materials may also be of lower cost than more
20 expensive piezoelectric materials, and may also be easier to machine or etch or mould channels and other surface features in that substrate. The rigid substrate may for example be provided by a conventional laboratory glass slide.

As noted above, the coupling of the piezoelectric substrate with the rigid secondary substrate may be achieved by means of a fluid coupling layer. The
25 fluid may be a moderate viscosity fluid such as H₂O, although the use of other fluids of lower and higher viscosities is also envisaged. The fluid coupling layer separates the opposing surfaces of the substrates such that there is no direct physical contact between the substrates.

The use of a fluid coupling layer located between the two substrates when
30 the secondary substrate overlays the piezoelectric substrate allows the SAW wave to be distributed to the secondary substrate with minimal loss of energy. The effect of any surface imperfections on the opposing piezoelectric and secondary substrates on the transfer of the SAW wave will be minimised or

eliminated by the use of this fluid coupling layer. It is noted that the use of any solid layer such as a solidified adhesive layer as a coupling between the piezoelectric substrate and secondary substrate may act to absorb and suppress the SAW wave preventing its distribution to the secondary substrate, since almost
5 all adhesives have extremely high acoustic losses.

Another advantage of the use of a fluid coupling layer is that the secondary substrate may not be permanently attached to the piezoelectric substrate. This means that relatively inexpensive secondary substrates as simple as a glass slide would be used and then discarded while the relatively expensive piezoelectric
10 substrate and wave generation means can be reused.

The piezoelectric substrate itself may be conventionally formed from Lithium Niobate (LiNbO_3). Other types of piezoelectric material may also be used as it is possible to induce waves in polycrystalline piezoelectric material including barium titanate (BaTiO_3), lead zirconium titanate (PZT or PbZrO_3 , often used with
15 dopants to improve performance), zinc oxide (ZnO), aluminium nitride (AlN), and single crystal materials like lithium tantalate (LiTaO_3), quartz, langasite ($\text{La}_3\text{Ga}_5\text{SiO}_{14}$), and gallium orthophosphate (GaPO_4) (this may be applicable for high temperature applications; the coupling fluid could then be a metal, and permit us to make high-temperature atomized particles of caustic solutions).

20 The wave generation means may include at least one interdigital electrode deposited on the piezoelectric substrate, and electrical supply means for applying an RF input into the electrode. It is however also envisaged that other types of transducers could be used to generate the SAW wave or other waves in the piezoelectric substrate.

25 The piezoelectric substrate may be elongate in shape having opposing ends, and at least one said interdigital electrode may be located at one end thereof. Preferably, an interdigital electrode can be provided at opposing ends of the piezoelectric substrate. The secondary substrate and fluid coupling layer may then be located over the piezoelectric substrate between the electrodes. These
30 interdigital electrodes will therefore be located away from and will not be covered by the secondary substrate. Application of an RF input into the electrode(s) will result in a SAW wave being generated along the length of the piezoelectric substrate. The fluid coupling layer may be in the form of a liquid droplet upon

which the secondary substrate is overlaid. The liquid droplet may be located away from the electrodes. It is to be appreciated that the use of the fluid coupling layer means that the secondary substrate does not need to be aligned perfectly parallel to the surface of the piezoelectric substrate.

5 In another possible arrangement, at least one interdigital electrode may be located under the secondary substrate and therefore at least partially within the fluid coupling layer. Preferably a number of said electrodes may be located under the secondary substrate. This arrangement which will be subsequently described in more detail, is particularly useful when microfluidic channels are provided in the
10 secondary substrate, and different SAW waves are distributed along different sections of the channels.

According to another aspect of the present invention, there is provided a method of synthesizing zeolite nanocrystals using the above described microfluidic system.

15 The method includes applying on the working surface a liquid droplet of a solution predominantly comprising NaOH, H_3PO_4 , silica and sodium aluminate, and a gelling polymer, with ethanol and/or water as solvent, and applying a SAW vibration to the working surface sufficient to cause atomisation of the fluid droplet thereby leading to the generation of said zeolite nanocrystals.

20 According to yet another aspect of the present invention, there is provided a method of analysing fluid using a microfluidic system as described above.

The secondary substrate may be provided with microfluidic channels through which fluid can be delivered, and at least one electrode may be provided under the secondary substrate on the piezoelectric substrate to thereby apply
25 waves to fluid passing through those channels. Preferably at least two supply channels may be provided, the supply channels coming together at a mixer zone. Reactants passing through each supply channel may be mixed at the mixer zone by means of an interdigital electrode located near the zone and generating a SAW wave in that mixer zone to thereby facilitate mixing and/or reacting of the
30 reactants. Following the mixing zone may be a test zone where the mixed reactants can be analysed. Integrated circuit devices may be located on the secondary substrate to analyse the mixed reactants within the channel of the test zone. Opposing electrodes on either side of the test zone channel may apply a

SAW wave to the test zone to facilitate the reaction therein. Downstream from the test zone may be provided a steering device provided by a further electrode provided near a channel junction which separates into at least two discharge channels. The electrode may generate a SAW wave as required to allow the reacted product to pass along one discharge channel where the reacted result is positive, or along another discharge channel acting as a waste channel when the reacted product is unacceptable.

It will be convenient to further describe the invention with respect to the accompanying drawings which illustrate preferred embodiments of the microfluidic system according to the present invention. Other arrangements of the invention are possible, and consequently, the particularity of the accompanying drawings is not to be understood as superseding the generality of the preceding description of the invention.

In the drawings:

Figure 1 is a plan view of a first preferred embodiment of a microfluidic system according to the present invention;

Figure 2 is a partial side view of the microfluidic system of Figure 1 showing its application in caustic solution atomisation;

Figure 3a is a partial side view of a second preferred embodiment of the microfluidic system according to the present invention; and

Figure 3b is a partial plane view of the microfluidic system of Figure 3a.

Referring initially to Figures 1 and 2, the microfluidic system according to a first preferred embodiment of a microfluidic system according to the present invention includes an elongate piezoelectric substrate 1 having an upper surface 3. Wave generation means in the form of Interdigital electrodes 5 are deposited on the surface 3 at opposing ends of the piezoelectric substrate 1. A radio frequency (RF) input 7 is provided to each of the interdigital electrodes 5. This pulse excitation of the interdigital electrodes 5 results in a surface acoustic wave (SAW) wave 9 within the piezoelectric substrate of a surface 3. Depending on the configuration of the interdigital electrodes 5 and the piezoelectric substrate 1, the SAW wave may be either a standing or a travelling wave generated within the upper surface 3. The frequency of the pulse excitation can typically be in the

order of between 10 to 1000 MHz, although this frequency can vary depending on the application of the microfluidic system.

Located above the upper surface 3 is a rigid secondary substrate 11, with a liquid droplet 13 being provided between the piezoelectric substrate upper surface 3 and the secondary substrate 11 to provide a fluid coupling layer 13 therebetween. This enables the SAW waves generated in the piezoelectric substrate 1 to be distributed to the working surface 12 of the secondary substrate 11 as best shown in Figure 2. The secondary substrate 11 is formed of relatively chemically inert material such as glass. This enables fluid droplets 15 of caustic solution to be supported on the working surface 12 of the secondary substrate 11. This caustic solution would otherwise corrode the surface of the piezoelectric substrate 1 if applied directly on to that surface 3. Figure 2 illustrates a particular application of the microfluidic system according to the present invention where fluid can be atomised mechanically due to the vibration of the working surface 12 when the SAW wave is distributed to the secondary substrate 11.

The microfluidic device according to the present invention may be used to synthesize aluminosilicate or silicoaluminophosphate zeolite nanocrystals. Zeolites are used in many industrial applications, for example in the production of gas separation membranes where the zeolite is embedded in a polymer matrix. It is at present difficult to synthesize zeolite nanostructures using conventional techniques. The microfluidic system according to the present invention however facilitates the production of zeolite nanocrystals. This is achieved by using the system to atomise fluid droplets 15 of a caustic solution predominantly comprising NaOH, H₃PO₄, silica and sodium aluminate, and a gelling polymer, with ethanol and/or water as solvent. These fluid droplets 15 can be applied to the working surface 12 of the secondary substrate 11 which, being chemically inert, will not be corroded by that caustic fluid. High frequency electric fields of 10 MHz to as much as 2 GHz may be applied to the electrodes 5 in order to induce exceptionally shallow (~5 wavelengths) mechanical SAW vibrations along the piezoelectric substrate surface 3. Placement of a liquid droplet 15 upon the working surface 12 permits transmission of this acoustic energy into the droplet which appears at the droplet's surface as a capillary wave. With sufficient acoustic power input into the droplet, usually over 0.5W and dependent upon the

viscosity of the droplet and its size, the droplet is atomized by generation of small particles that form via breakage and separation at each of the capillary wave peaks.

The advantages of SAW atomization are its high energy density, efficient
5 piezoelectric energy conversion, and straightforward particle size control method. Moreover, the power requirement, typically a continuous 2-5 W, is low, allowing the design of a compact device. Recent studies by the Applicant have demonstrated the possibility of generating copious (several mL/min) amounts of nanodrops of 10–1000 nm in diameter. The wavelength of the capillary wave in
10 the fluid is directly related to the wavelength of the SAW radiation along the substrate surface, and so the atomized droplet size is controlled by the frequency of the SAW and the physical characteristics of the fluid set atop the working surface 12.

Figures 3a and 3b show an alternative preferred embodiment of the
15 microfluidic system of the present invention. The use of the same reference numerals for corresponding features is used in the description of this alternative arrangement for clarity reasons. This second preferred embodiment also includes a piezoelectric substrate 1 having an upper surface 3. A secondary substrate 11 is also located above the upper surface 3, with a fluid coupling layer
20 13 being provided therebetween. The principal differences are that a plurality of interdigital electrodes are located under the secondary substrate 11, and a series of channels 17 are provided within the working surface 12 of the secondary substrate 11.

Referring specifically to Figure 3b the microfluidic channels 17 include a
25 pair of supply channels 19a and 19b through which different reactants can be separately supplied. The supply channels 19a, 19b merge at a mixer zone 21 where the different reactants can mix and react. Located adjacent to the mixer zone 21 is a first curved interdigital electrode 23 which distributes a SAW wave through the fluid coupling 13 to the mixer zone to facilitate mixing of the reactants
30 therein. The mixed reactants then move to a test zone 25 located downstream from the mixer zone 21. A test arrangement (not shown) can be located on the secondary substrate 11 adjacent the test zone 25 to enable analysis of the mixed reactants located within the test zone 25. For example, IC devices could be

readily secured to the working surface 12 adjacent the test zone 25. Opposing interdigital electrodes 27 are located on opposing sides of the test zone 25 to thereby allow for the distribution of a further SAW wave to the test zone 25 to facilitate the reaction of the mixed reactants. Downstream from the test zone 25 are two discharge channels 29a and 29b. A second curved interdigital electrode 31 is located adjacent the junction 28 of the two discharge channels 29a, 29b. The secondary interdigital electrode can be selectively actuated to direct the next reactants through one of the discharge channels 29a where the reacted results is positive or along the other discharge channel 29b when the reacted product is unacceptable.

Modifications and variations as would be deemed obvious to the person skilled in the art are included within the ambit of the present invention as claimed in the appended claims.

CLAIMS:

1. A microfluidic system including:
a piezoelectric substrate;
a wave generation means for generating a wave in the piezoelectric
5 substrate; and
a rigid secondary substrate coupled to said piezoelectric substrate and
providing a working surface for the system, the secondary substrate being formed
of a relatively chemically inert material;
the coupling of the piezoelectric substrate with the rigid secondary
10 substrate being provided by a fluid coupling layer located between the
piezoelectric substrate and the secondary substrate overlaying the piezoelectric
substrate.
2. A microfluidic system according to claim 1, wherein the wave generation
means generates a surface acoustic wave (SAW) in the surface of the
15 piezoelectric substrate.
3. A microfluidic system according to claim 1 or 2, wherein the wave
generation means includes at least one interdigital electrode located on the
piezoelectric substrate.
4. A microfluidic system according to any one of the preceding claims,
20 wherein the secondary substrate is provided with microfluidic channels through
which fluid can be delivered, and at least one electrode is provided under the
secondary substrate on the piezoelectric substrate to thereby apply waves to fluid
passing through those channels.
- 25 5. A microfluidic system according to claim 4, wherein at least two supply
channels are provided, the supply channels coming together at a mixer zone
where fluid passing through each supply channel can be mixed by means of an
interdigital electrode located near the mixer zone generating a SAW wave in that
mixer zone, a test zone downstream of the mixer zone where the mixed and/or

reacted fluids can be tested, with opposing electrodes located on either side of the test zone channel to apply a SAW wave to the test zone to facilitate the reaction therein, and a steering device provided by a further steering electrode provided near a channel junction located downstream from the test zone which
5 separates into at least two discharge channels, the steering electrode selectively generating a SAW wave as required to allow the reacted fluid to pass along one discharge channel where the reacted result is positive, or along another discharge channel acting as a waste channel when the reacted product is unacceptable.

10 6. A method of synthesizing electrolyte nanocrystals using the microfluidic system as claimed in any one of the preceding claims:

7. A method according to claim 6, including applying on the working surface a liquid droplet of a solution predominantly comprising NaOH, H₃PO₄, silica and sodium aluminate, and a gelling polymer, with ethanol and/or water as solvent,
15 and applying a SAW vibration to the working surface sufficient to cause atomisation of the fluid droplet thereby leading to the generation of said zeolite nanocrystals.

8. A method of analysing fluid using a microfluidic system as claimed in claim 4 or 5.

20 9. A method according to claim 8, including supplying different reactant fluids through said channels and mixing and reacting said reactant fluids therein, the at least one electrode applying a wave to at least a section of said channels.

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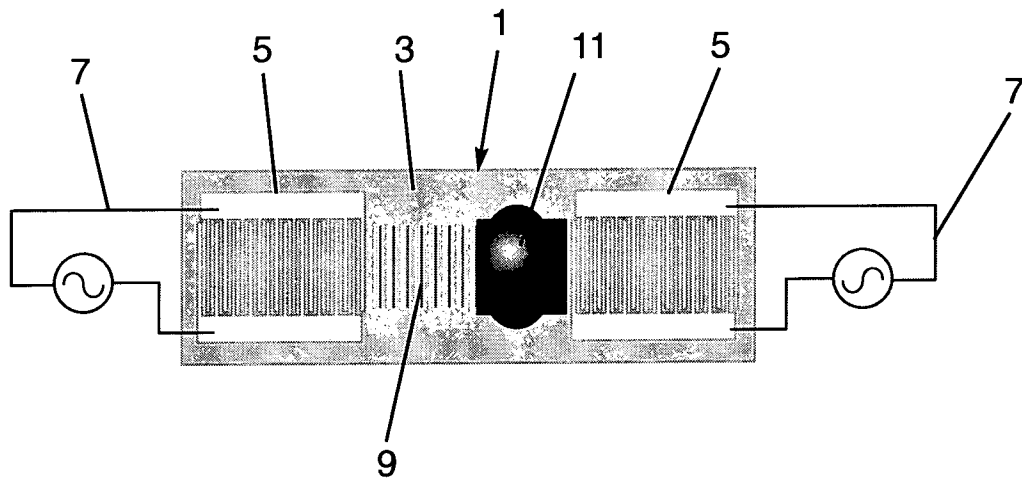


Figure 1

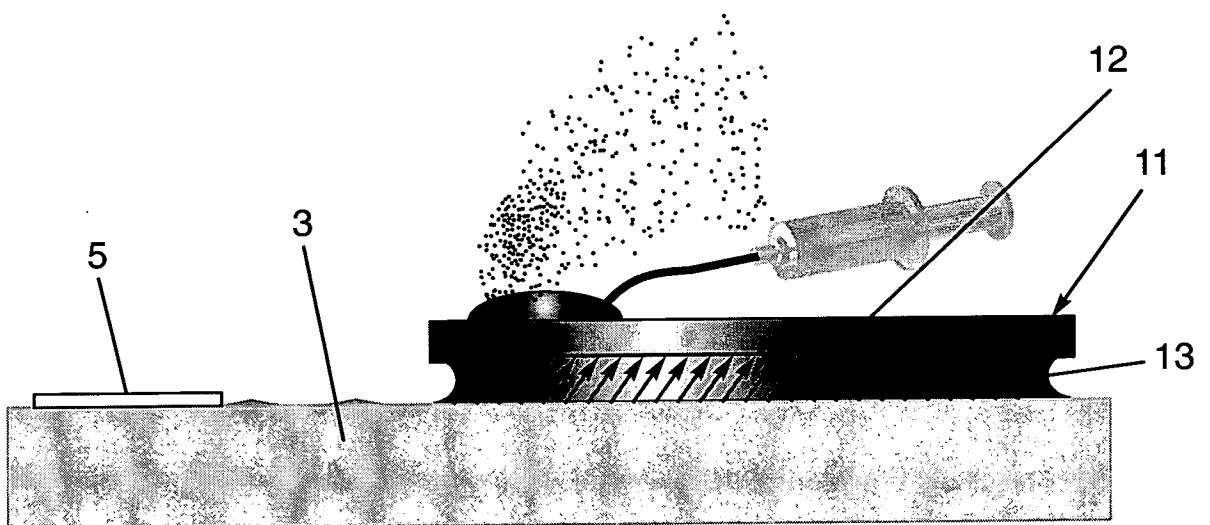


Figure 2

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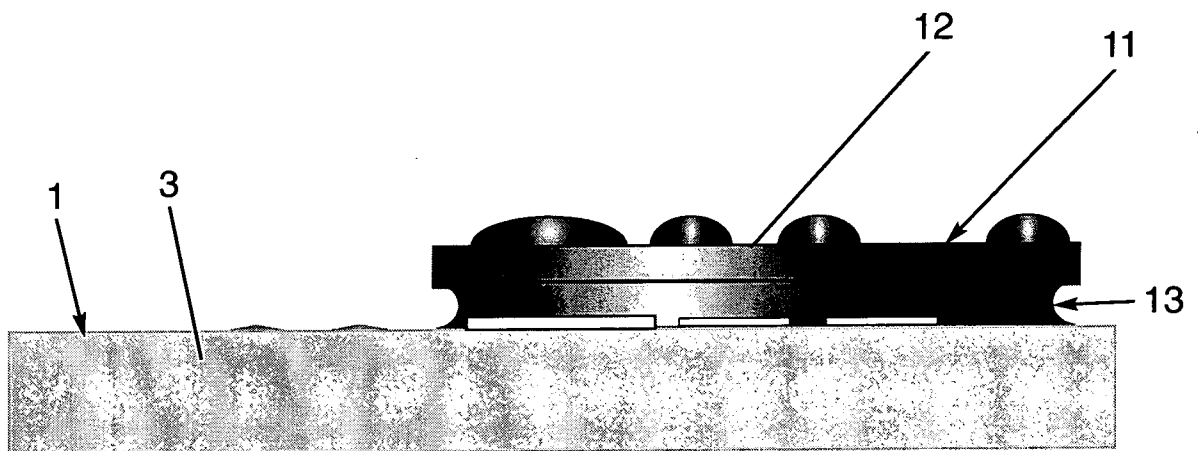
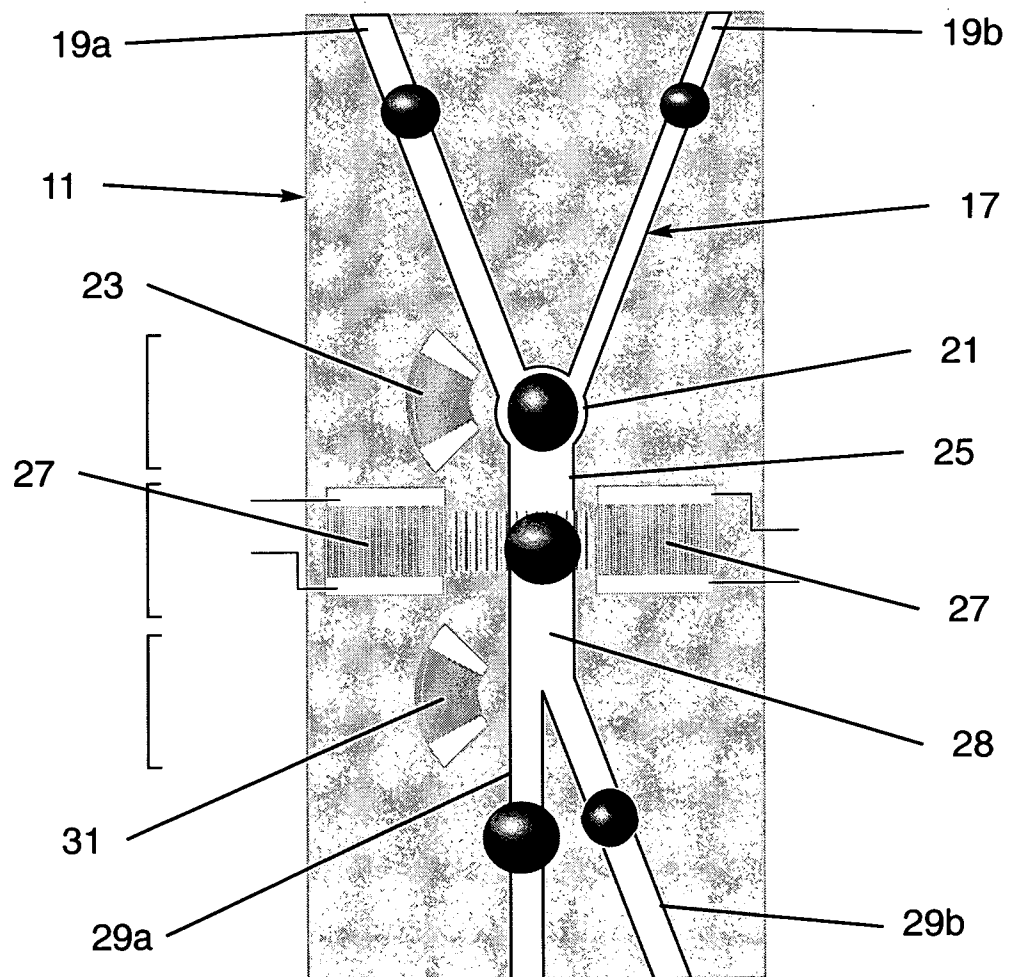


Figure 3a



INTERNATIONAL SEARCH REPORT

International application No.
PCT/AU2007/000575

A. CLASSIFICATION OF SUBJECT MATTER

Int. Cl.

B81B 7/02 (2006.01)**C30B 29/34** (2006.01)**G01N 1/28** (2006.01)**B06B 1/06** (2006.01)**C30B 30/06** (2006.01)**H01L 41/09** (2006.01)

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)

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

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

DWPI Keywords: microfluid, micro total analysis, biochip, biomems, utas, lab on chip, etc; piezo, pzt; acoustic, ultrasonic, wave, vibration; couple, intermediate; nanocrystal, zeolite, quantum dot; interface

GOOGLE Keywords: acoustic, piezoelectric, microfluidic, nanoparticle, atomization

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	SRITHARAN et al. 'Acoustic mixing at low Reynold's numbers', Applied Physics Letters, vol 88, article 054102, 2 February 2006 See pages 1-2 and fig 2-3	1-9
X	US 2006/0024206 A1 (SINHA et al) 2 February 2006 See paragraphs 4-5, 23-26 and fig 1a and 6	1, 2, 4, 6-9
X	US 2002/0022261 A1 (ANDERSON et al) 21 February 2002 See paragraphs 114, 205-206 and fig 7a	1, 2, 6, 7

☒ Further documents are listed in the continuation of Box C☒ See patent family annex

* Special categories of cited documents:		
"A" document defining the general state of the art which is not considered to be of particular relevance	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention	
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone	
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art	
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family	
"P" document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search
6 June 2007

Date of mailing of the international search report
18 JUN 2007

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INTERNATIONAL SEARCH REPORT

International application No.

PCT/AU2007/000575

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
P, X	WO 2006/051153 A2 (NANOLAB SYSTEMS OY) 18 May 2006 See pages 4, 9, 15-16 and fig 8-9	1, 2, 4, 6, 8, 9
P, X	TAN et al. 'Microparticle collection and concentration via a miniature surface acoustic wave device', Lab on a Chip, 18 April 2007, vol. 7, pages 618-625 See whole document	1-4, 8
A	PINGALI et al. 'Silver nanoparticles from ultrasonic spray pyrolysis of aqueous silver nitrate', Aerosol Science and Technology, 2005, vol. 39, pages 1010-1014 See whole document and fig 2	6, 7
A	US 2004/0066703 A1 (SPAREY-TAYLOR et al.) 8 April 2004 See whole document	1-9
A	US 2004/0257906 A1 (SCRIBA et al.) 23 December 2004 See whole document	1-9
A	STROBL et al. 'Planar microfluidic processors', IEEE Ultrasonics Symposium, 2002, pages 255-258 See whole document	1-9
A	KUROSAWA et al. 'Surface acoustic wave atomizer with pumping effect', Micro Electrical Mechanical Systems, IEEE Proceedings, 2 February 1995, pages 25-30 See whole document	1-9

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/AU2007/000575

This Annex lists the known "A" publication level patent family members relating to the patent documents cited in the above-mentioned international search report. The Australian Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

Patent Document Cited in Search Report			Patent Family Member			
US	2006024206	NONE				
US	2002022261	AU 64049/96	EP 0843734	EP	1304388	
		US 5856174	US 5922591	US	6043080	
		US 6168948	US 6197595	US	6326211	
		US 6830936	US 2001036672	US	2005100946	
		US 2005202504	US 2005250199	US	2006246490	
		WO 9702357				
WO	2006051153	NONE				
US	2004066703	AU 2003279814	WO 2004030800			
US	2004257906	EP 1420875	WO 03018181			
Due to data integration issues this family listing may not include 10 digit Australian applications filed since May 2001.						
END OF ANNEX						