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(54) **LIQUID EVAPORATOR**

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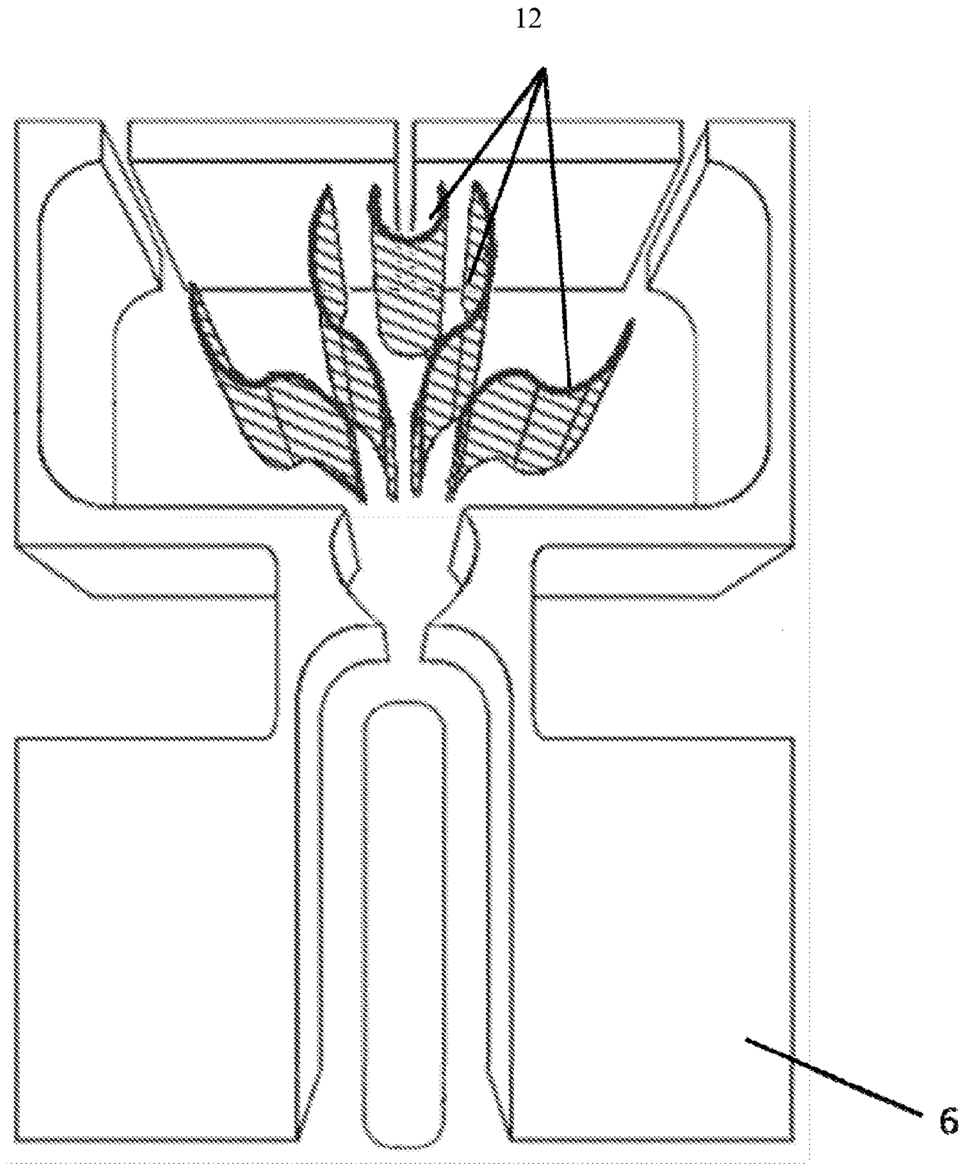
(57) **ABSTRACT**

The present invention relates to a liquid evaporator and to a method for evaporating liquids.



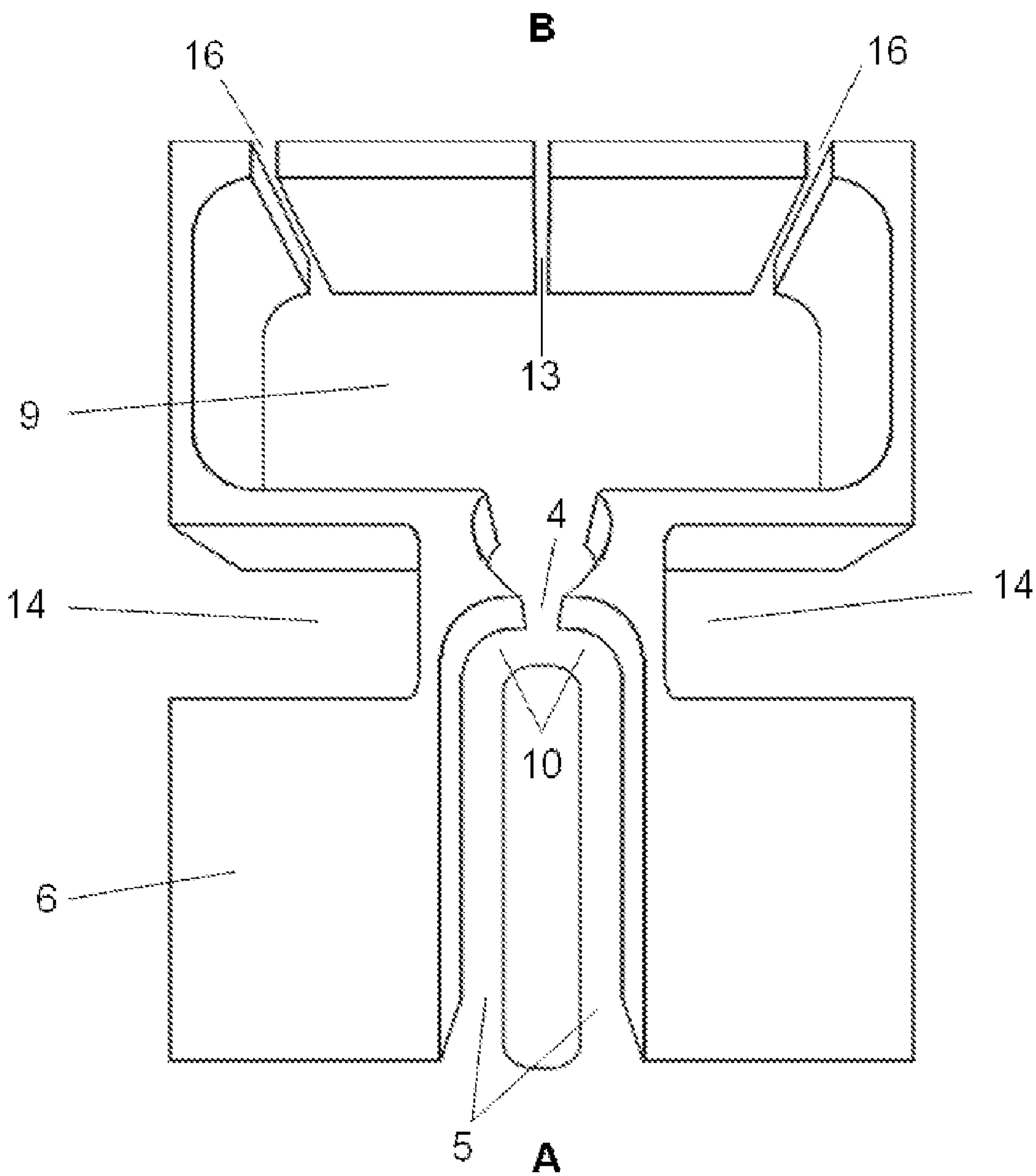


Fig. 1

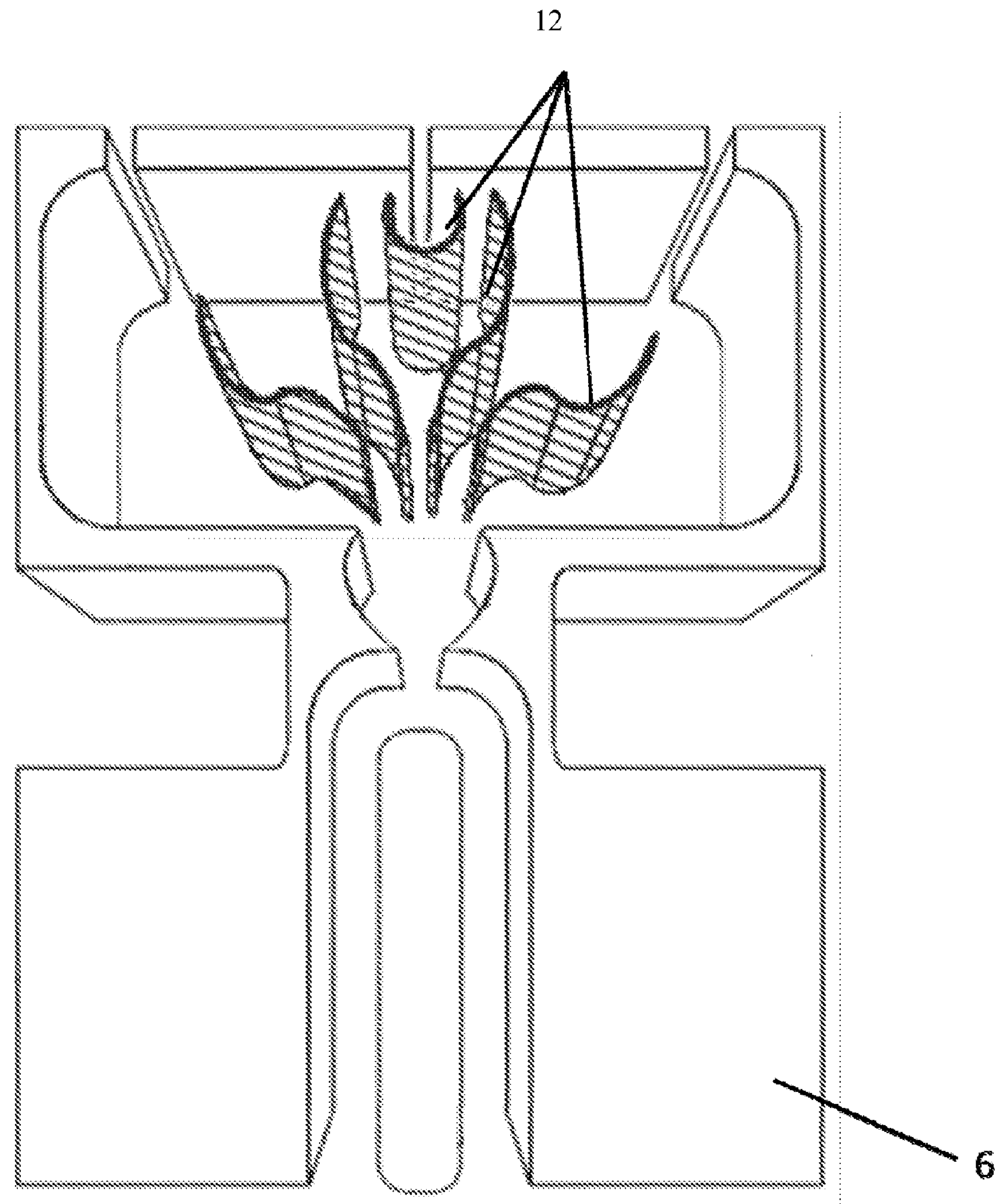


Fig. 2

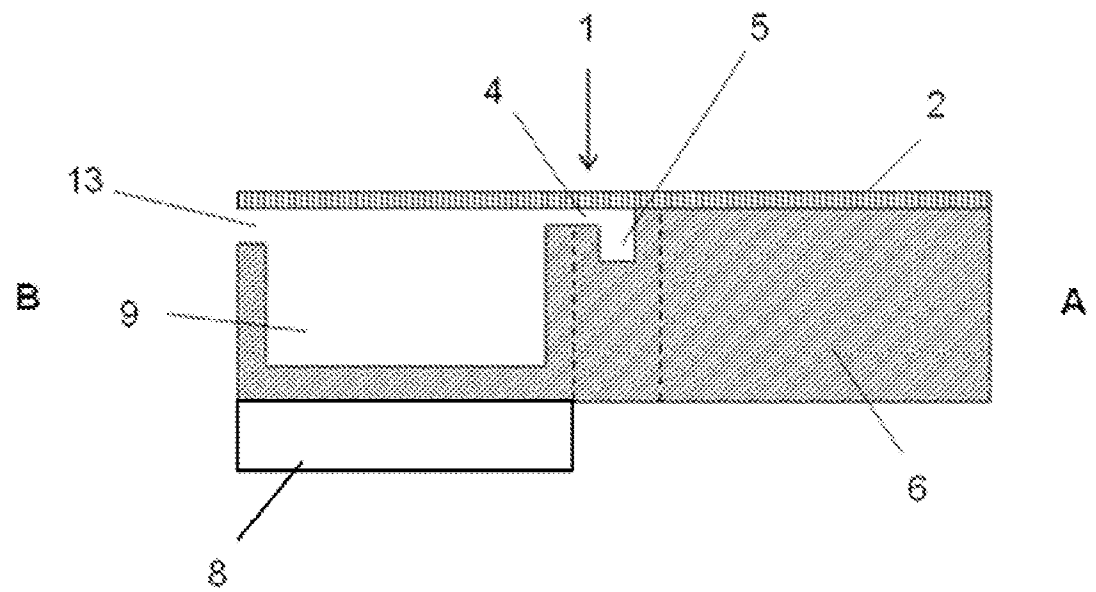


Fig. 3

LIQUID EVAPORATOR

[0001] The present invention relates to a liquid evaporator and to a method for evaporating liquids.

[0002] Liquid evaporators are used for converting liquids into the gas phase and are employed in various applications.

[0003] U.S. Pat. No. 7,618,027B2 discloses, for example, a liquid evaporator for generating a highly pure gas with a low vapour pressure, the gas being used in the field of microelectronics.

[0004] WO05/016512A1 discloses, for example, a liquid evaporator which can be used in a method for removing a volatile compound from a substance mixture.

[0005] Liquid evaporators also have widespread use in analysis, where a sample quantity of a liquid to be analysed is first converted into the gas phase in order to make it accessible for an analysis method. U.S. Pat. No. 7,309,859B2 discloses, for example, a liquid evaporator which can be used in an ion source for mass spectrometers.

[0006] Particularly in the field of analysis, special requirements are placed on a liquid evaporator. In the case of substance mixtures for quantitatively reliable analysis, for example, it is important for the concentrations of all components of the substance mixture in the gas phase to be identical to the concentrations in the liquid. To this end, it is necessary to carry out the evaporation of the sample volume completely. In order to avoid recondensation, the gaseous sample then needs to be delivered, for example, to the analysis system above the highest evaporation temperature of the components involved.

[0007] To date, liquid evaporators have preferably been configured as constantly heated systems to which liquid samples are supplied continuously in small quantities. In order to ensure the necessary complete, and as far as possible simultaneous, evaporation of the components which have different evaporation temperatures, a high heat capacity of the evaporator is generally necessary, together with a high thermal mass. This entails a high energy demand and—owing to the in general spatially extended structure of the evaporator—a correspondingly high power demand and concomitantly sometimes also long dead times between sampling and evaporation.

[0008] Owing to the increasing performance of modern analysis systems, the sample quantity required for an analysis has constantly decreased over the course of time. For example, micro mass spectrometers are known (see for example WO08/101669A1) which can function with minimal sample quantities.

[0009] The reduction of the required sample quantity is on the one hand advantageous. Smaller sample quantities mean, for example, a shorter evaporation time and therefore a higher temporal resolution with which changes in sample composition can be recorded.

[0010] On the other hand, the liquid evaporator needs to be adapted to the small sample quantity in order to be able to fully exploit the advantages of a smaller sample quantity.

[0011] On the basis of the known prior art, the object therefore arises to provide a liquid evaporator and a liquid evaporation method, with which small volumes of liquids can be evaporated reliably. The sample volume to be evaporated is in this context preferably less than 100 μl, particularly preferably less than 10 μl, more particularly preferably less than 1 μl.

[0012] The sample quantity evaporated should be representative of the liquid from which it is taken. The liquid evaporator should be economical to manufacture and operate, have a low energy demand for the evaporation and ensure rapid evaporation. The liquid evaporator should either have self-cleaning capabilities, so that it is even possible to evaporate liquids which contain dissolved substances that may possibly leave a deposit, or be configured as a disposable article.

[0013] According to the invention, this object is achieved by a method for evaporating at least a part of a liquid according to Claim **1**, and by a liquid evaporator according to Claim **4**, which is formed in order to carry out the method. Preferred embodiments may be found in the dependent claims.

[0014] The present invention therefore firstly provides a method for evaporating at least a part of a liquid, characterized in that a liquid is fed through a channel past an opening, the opening leading to a vapour chamber which is maintained at a temperature above the evaporation temperature of the liquid, and the liquid is heated in the region of the opening by electromagnetic radiation so that at least a part of the liquid evaporates and the vapour enters the heated vapour chamber.

[0015] The present invention secondly provides a liquid evaporator comprising at least

[0016] a body in which a channel and a chamber, which are connected to one another through an opening, are formed, the channel being configured so that a liquid can be fed past the opening and an evaporation region adjacent to the opening, and optionally including the opening, can be irradiated with electromagnetic radiation,

[0017] means for heating the chamber.

[0018] The method according to the invention and the evaporator according to the invention use the energy of electromagnetic radiation in order to evaporate a sample quantity of a liquid by local heating. To this end, a liquid is passed through a channel which leads past an opening into a chamber. The region of the opening is accessible for electromagnetic radiation, i.e. it can be irradiated with electromagnetic radiation. In this region—also referred to below as the evaporation region—the liquid is locally heated by means of electromagnetic radiation to such an extent that a sample quantity evaporates. The evaporated sample quantity passes through the opening into a chamber—also referred to below as the vapour chamber—which can be heated to a temperature above the evaporation temperature of the liquid, so that the vapour does not condense in the chamber.

[0019] The electromagnetic radiation is preferably supplied to the evaporation site from outside the liquid evaporator. The evaporation site is therefore preferably provided with a cover which is at least partially transparent for the electromagnetic radiation being used. An at least partially transparent cover is intended to mean a cover which preferably transmits a majority of the electromagnetic radiation and absorbs and/or reflects only a small part, so that a majority of the incident energy reaches the evaporation site and is available there for heating a sample quantity. A high transparency and therefore low absorption of the cover also have the effect that the cover itself is not heated.

[0020] Preferably, the evaporation region is configured so that it absorbs a high proportion of the electromagnetic radiation and converts it into heat. For example, the inner walls of the channel may consist of a material which absorbs a majority of the incident energy and converts it into heat. It is likewise conceivable for the inner walls of the channel to be coated in the evaporation region with a material which has a high absorption coefficient for the radiation being used. In both of these cases, by means of the evaporator it is even possible to evaporate liquids which themselves have only a low absorption coefficient for the radiation being used; the heating takes place indirectly. The evaporation region, when it is adapted for indirect heating of the liquid, will also be referred to below as the absorber.

[0021] Advantageously, however, the radiation source used is matched to the liquid to be evaporated so as to allow direct heating of the liquid. Direct heating has the advantage that the environment of the liquid is heated only slightly, and detrimental effects on the analysis due to a heated environment (lower temporal resolution, contamination, damage, etc.) are thus minimized.

[0022] A laser beam is preferably used for the irradiation. A pulsed, focused laser beam is particularly preferably used. The laser pulse length is preferably selected so that the thermal time constants of the absorber and/or the liquid to be evaporated are long compared with this pulse length. This means that even a single pulse is sufficient to evaporate a sample quantity.

[0023] The quantity of liquid evaporated may be varied by varying the length of the laser pulse and by varying its power. For the evaporation of larger quantities, a pulse sequence may also be used. Since the instant of the evaporation can be established very accurately, the vapour generation can be synchronized with sampling in an analyser so that a correlation measurement with increased measurement sensitivity is possible. Furthermore, this method allows segments of a sample flow to be evaporated and analysed in a controlled way, so that in particular even sample compositions which vary greatly as a function of time or, for example, unmixed or undissolved components carried by the liquid (emulsions, cells) can be recorded in a defined or selective way.

[0024] The laser beam is preferably supplied to the evaporation region through a cover, which is at least partially transparent for the laser beam, by means of free-beam or fibre optics.

[0025] The transition from the channel to the vapour chamber is configured so that, because of the capillary forces prevailing in the opening, the liquid cannot flow freely into the chamber. Suppression of the liquid egress is preferably achieved by a sufficiently large difference between the cross section of the chamber dimension at the position of the opening and the length of the interface with the channel and its cross section. As an alternative or in addition, the inner walls of the channel and the chamber may be provided, at least in the opening region, with layers having different surface energies. If the liquid to be evaporated is an aqueous solution, for example, the opening region of the channel may be coated hydrophilically and that of the chamber may be coated hydrophobically.

[0026] In order to permit sampling from the middle of the channel, particularly in the case of small cross sections and therefore non-turbulent laminar flow, the channel preferably has a curvature in the region of the evaporation. The curvature leads to different flow rates in the inner and outer curve regions. Dean vortices are generated, which lead to a flow perpendicular to the flow direction and convey liquid elements from the middle of the channel to the channel edge in the opening region.

[0027] Since the briefly heated position is constantly flushed with the following liquid, it is also possible to achieve the effect that solids possibly precipitated during the evaporation are either redissolved or mechanically removed by the liquid flow and transported away. The Dean vortices occurring in the curvature region of the channel assist this self-cleaning process.

[0028] The vapour chamber has a gas outlet, through which the vapour can leave the liquid evaporator according to the invention. Besides the gas outlet, the chamber may be provided with further connections which, in particular, allow evacuation and optionally also flushing of the chamber between the sampling intervals, in order to avoid cross-contamination of samples from one evaporation process to the next.

[0029] As an alternative, this may also respectively be done in a controlled way after a plurality of evaporations, in order to increase the available gas quantity and/or its pressure for the injection or to permit averaging over a plurality of sample volumes directly before the analysis.

[0030] The vapour chamber may be heated by means of a heating element, which is preferably operated electrically.

[0031] In order to keep the vapour uniformly at a high temperature, preferably lamellar structures made of a thermally conductive material may additionally extend through the vapour chamber, which preferably is locally heated by surface contact heating, these structures likewise being heated by the heating device via thermal conduction.

[0032] These structures also facilitate the evaporation of droplets which have been entrained by the vapour from the channel into the chamber.

[0033] The structures are preferably applied so that they prevent particles from passing through the gas outlet (see FIG. **2**), i.e. they preferably shield the opening and the gas outlet from one another.

[0034] Since the input of energy for the evaporation preferably takes place optically, i.e. contactlessly, and the heating of the chamber does not need to be integrated directly therein, the evaporation system is simple to produce and in principle can also be replaced easily in the event of contamination. In a preferred embodiment, the liquid evaporator according to the invention is configured as a disposable article.

[0035] The body of the liquid evaporator according to the invention, in which the channel and chamber are formed, may be configured in one piece or a plurality of pieces. It is preferably configured in one piece.

[0036] The liquid evaporator is preferably a microsystem, the structures of which are produced by means of microfabrication techniques.

[0037] The production of structures in microsystems is known to the person skilled in the art of microsystem technology. Microfabrication techniques are, for example, described and illustrated in the book "Fundamentals of Microfabrication" by Marc Madou, CRC Press Boca Raton Fla. 1997 or in the book "Mikrosystemtechnik für Ingenieure" [Microsystem technology for engineers] by W. Menz. J. Mohr and 0. Paul, Wiley-VCH, Weinheim 2005. A more detailed description of silicon-on-silicon technology may for example be found in Q.-Y. Tong, U. Gösele: *Semiconductor Wafer Bonding: Science and Technology;* The Electrochemical Society Series, Wiley-Verlag, New York (1999). With regard to glass-on-glass technology, reference may be made by way of example to the following publications: J. Wie et al., *Low Temperature Glass-to-Glass Wafer Bonding,* IEEE Transactions on advanced packaging, Vol. 26, No. 3, 2003, pages 289-294; Duck-Jung Lee et al., *Glass-to-Glass Anodic Bonding for High Vacuum Packaging of Microelectronics and its Stability,* MEMS 2000, The Thirteenth

Annual International Conference on Micro Electro Mechanical Systems, 23-27 Jan. 2000, pages 253-258.

[0038] Microsystem technologies are fundamentally based on the structuring of silicon and/or glass substrates with a high aspect ratio (for example narrow trenches (~μm) of great depth (~100 μm)) with structuring accuracies in the micrometre range using wet chemical, preferably plasma etching processes combined with sodium-containing glass substrates adapted in terms of their thermal expansion coefficient (for example Pyrex®), which are provided with simple etched structures and preferably connected to one another with a hermetic seal directly by so-called anodic bonding, or alternatively with a thin Au layer functioning as a solder alloy (AuSi).

[0039] Metal structures with a high aspect ratio can be produced by electrolytic growth in thick photoresists (>100 μm) with comparable accuracy (UV-LIGA). By using thin-film technologies such as high vacuum evaporation and sputtering, PVD processes or chemical vapour deposition (CVD processes) preferably in a plasma, in combination with photolithography and etching techniques, functional layers such as metallizations, hydrophobic or hydrophilic surfaces and functional elements such as valve seals and diaphragms, heating elements, temperature, pressure and flow sensors can be integrated on these substrates in a fully process-compatible technology.

[0040] The structures of the liquid evaporator according to the invention are preferably produced in a silicon-on-glass technology, silicon being used for the body and glass being used for the transparent cover. This combination, preferably connected hermetically by anodic bonding, allows highly accurate structuring of the various components of the system, particularly in silicon (photoetch technology, DRIE, coating). Silicon is chemically and thermally stable like glass, and in contrast to glass it is a good thermal conductor with low heat capacity (heated chamber with uniform temperature) and a good optical absorber for conventional laser wavelengths. The heat losses by dissipation through the glass substrate are low.

[0041] The combination of silicon and glass makes it possible to achieve local input of the optical energy into the channel edge as well as thermal decoupling of the channel and the vapour chamber. For thermal decoupling, the vapour chamber and the liquid sample channel are preferably separated by horizontal and vertical incisions in the highly thermally conductive body. Mechanical stability with low heat transfer is ensured by a transparent cover, for example made of glass or a polymer.

[0042] It is also conceivable to produce the liquid evaporator according to the invention from polymer materials, for example by means of injection moulding techniques. A composite material is preferably used for the body, for example a polymer in which carbon (carbon black, carbon nanotubes) is dispersed in order to increase the absorption of electromagnetic radiation and the thermal conductivity.

[0043] Owing to the conventional dimensions in microsystem technology, in the range of a few 10 to 100 μm, a system produced in this way is also especially suitable for analysing or producing small sample volumes (nl liquid, μl in the gas phase).

[0044] In order to evaporate such small sample quantities, only low laser energies in the mWs range are required even for liquids having a high enthalpy of vaporization, for example water, which are preferably delivered by standard semiconductor lasers, coupled via fibres or lenses, in the wavelength range of for example 400 nm to 980 nm even with the necessary short pulse durations (for example 1 W for 1 ms, 100 mW for 10 ms).

[0045] The sample volume to be evaporated is preferably less than 100 μl, particularly preferably less than 10 μl, more particularly preferably less than 1 μl.

[0046] The liquid evaporator according to the invention is preferably suitable as a sample evaporator in a microanalysis system. The present invention therefore also provides the use of the liquid evaporator according to the invention in a microanalysis system, particularly in a micro gas chromatograph or a micro mass spectrometer, as described for example in the article "*Complex MEMS: A fully integrated TOF micro mass spectrometer*" published in Sensors and Actuators A: Physical, 138 (1) (2007), pages 22-27.

[0047] The liquid evaporator according to the invention and the method according to the invention ensure the following points:

- **[0048]** simultaneous evaporation of liquids and liquid mixtures
- **[0049]** reliable evaporation of small volumes (preferably even <1 μl)
- **[0050]** short dead time between the evaporation of two samples
- **[0051]** rapid evaporation, so that a high temporal resolution can be achieved during the analysis
- **[0052]** evaporation of representative samples
- **[0053]** low energy requirement for the evaporation
- **[0054]** low equipment outlay
- **[0055]** even suitable for media having a high enthalpy of vaporization
- **[0056]** even suitable for media comprising dissolved solids (deposit)
- **[0057]** economical operation (production and replacement)
- **[0058]** low outlay on peripherals
- **[0059]** sampling synchronizable with the analysis of the sample (lock-in principle)
- **[0060]** doseability of the gas volume
- **[0061]** adjustability of the injection gas pressure
- **[0062]** averaging by means of synchronization and multiple pulses
- **[0063]** statistical selection (time window) of the samples to be averaged
- **[0064]** averaging in the gas space
- **[0065]** self-cleaning

[0066] The invention will be explained in more detail below with the aid of figures, but without being restricted thereto.

[0067] The following references are used in all the figures:

LIST OF REFERENCES

[0068] **1** electromagnetic radiation
[0069] **2** transparent cover
[0070] **3** liquid
[0071] **4** opening
[0072] **5** channel
[0073] **6** body
[0074] **7** vapour stream
[0075] **8** heating element
[0076] **9** vapour chamber
[0077] **10** curvature
[0078] **11** transverse flow
[0079] **12** lamellar structures

[0080] **13** gas outlet

[0081] **14** incision

[0082] **15** connection

[0083] FIG. **1** shows a perspective representation of a liquid evaporator according to the invention from above. The liquid evaporator comprises a body **6**, in which a channel **5** and a chamber **9** are formed. The channel **5** and the chamber **9** are connected to one another through an opening **4**. The channel **5** has a curvature **10** in the region of the opening **4**. A liquid is fed through the channel **5** past the opening **4**.

The transition from the opening **4** to the chamber **9** is configured so that, because of the capillary forces prevailing in the opening, the liquid cannot flow into the directly adjacent region **9**.

[0084] In the region of the opening, the liquid is irradiated by means of electromagnetic radiation and therefore heated. In the present case, the irradiation takes place from the direction of the observer (from above).

[0085] Owing to the irradiation, the liquid is heated and a part of it evaporates, this part entering the chamber **9** through the opening **4** as a vapour stream. Below the chamber **9**, there is a heating element by which the chamber can be heated (not shown in FIG. **1**; see FIG. **3**).

[0086] The vapour chamber **9** and liquid sample channel **5** are thermally decoupled by horizontal incisions **14** in the highly thermally conductive body **6**.

[0087] FIG. **2** shows the liquid evaporator of FIG. **1**, in the chamber **9** of which lamellar structures **12** are introduced. The lamellar structures are preferably heated by the heating element below the chamber **9** (not shown in FIG. **2**; see FIG. **3**) via thermal conduction.

[0088] FIG. **3** shows the liquid evaporator according to the invention of FIG. **1** in cross section along a straight line from A to B. Besides the components already described above in relation to FIG. **1**, a transparent cover **2** can be seen in FIG. **3** which extends over the entire body **6**. A heating element **8** is furthermore installed below the vapour chamber. The irradiation of the liquid takes place through the transparent cover, preferably by means of a focused laser beam **1**.

1. A method for evaporating at least a part of a liquid, said method comprising feeding said liquid through a channel past an opening, said opening leading to a vapour chamber which is maintained at a temperature above evaporation temperature of said liquid, and said liquid is heated in a region of the opening by electromagnetic radiation so that at least a part of the liquid evaporates and vapour enters the heated vapour chamber.

2. The method according to claim **1**, wherein said heating is carried out by a focused and pulsed laser beam.

3. The method according to claim **1**, wherein said liquid is a mixture.

4. A liquid evaporator comprising: at least

a body in which a channel and a chamber, which are connected to one another through an opening, are formed, said channel being configured so that liquid can be fed past the opening and there being an evaporation region adjacent to the opening, and optionally including the opening, and wherein said liquid can be irradiated with electromagnetic radiation, and

means for heating the chamber.

5. A liquid evaporator according to claim **4**, wherein said evaporation region comprises a cover which is at least partially transparent for said electromagnetic radiation being used.

6. A liquid evaporator according to claim **4**, wherein said channel, opening and chamber are configured so that a liquid flowing through said channel remains in said channel because of capillary forces and/or interfacial energies, and does not flow freely into the chamber.

7. A liquid evaporator according to claim **4**, wherein said channel has a curvature in a region of said opening, which generates Dean vortices in a flowing liquid.

8. A liquid evaporator according to claim **4**, further comprising

a gas outlet in said chamber,

connections in said chamber for evacuating and/or flushing the vapour chamber, and

lamellar structures in the chamber, which are connected in a thermally conductive fashion to said means for heating said chamber and which are optionally arranged to shield said gas outlet from said opening.

9. A liquid evaporator according to claim **4**, wherein said liquid evaporator is produced as a microsystem in a silicon-on-glass technology, silicon being used for the body and glass being used for the transparent cover.

10. The liquid evaporator according to claim **4**, which is capable of being used in a microanalysis system, optionally in a micro mass spectrometer or a micro gas chromatograph.

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