[0187] All silicon surfaces are oxidized to form silicon dioxide with a thickness that is controllable through choice of temperature and time of oxidation. All silicon dioxide surfaces are LPCVD coated with silicon nitride. The final thickness of the silicon dioxide and silicon nitride can be selected to provide the desired degree of electrical isolation in the device. A thicker layer of silicon dioxide and silicon nitride provides a greater resistance to electrical breakdown.

[0188] In situ Preparation of a Porous Polymer Monolith within a Microchip

[0189] The following is a general procedure for in situ preparation of a porous polymer monolith within the through-wafer channels and/or reservoir of a microchip electrospray device of the present invention. FIGS. **17**A-**17**B are used as illustration of these basic steps. A cleaned silicon substrate **200** is placed within a container along with a mixture **252** of monomers, porogens and initiator. Suitable monomers include styrene, acrylic acid and its esters, methacrylic acid and its esters, vinyl pyridine, maleate, vinylester, vinyl ether, and vinylalcohol derivatives, crosslinked with divinylbenzene, ethylene dimethacrylate or diacrylate, diethylene glycol dimethacrylate or diacrylate, divinylpyridine, bis-N-vinyl-2-pyrrolidone, N,N-methylene-bisacrylamide or bismethacrylamide, or trimethylolpropane trimethacrylate. Porogens include suitable solvents compatible with the polymerization process of the present invention. A compatible porogen solubilizes the monomer but not the polymer to create pores in the polymerized material. The system is purged with nitrogen to degas the mixture and remove excess oxygen. A coverplate **249** is placed on each side of the substrate to seal the mixture within the reservoirs **232** and through-substrate channels **234**. The system is heated to a temperature suitable for polymerization of the mixture or is polymerized using UV light if a UV sensitive initiator is used to form the integrated device containing polymer monolith **254**, as shown in **FIG. 17B**. The coverplates **249** are removed.

[0190] Alternately, after fabrication of multiple polymer monolith-electrospray devices on a single silicon wafer, the wafer can be diced or cut into individual devices. This exposes a portion of the silicon substrate **200** as shown in the cross-sectional view of **FIG. 18** on which a layer of conductive metal **247** can be deposited using well known thermal evaporation and metal deposition techniques.

[0191] The fabrication method confers superior mechanical stability to the fabricated electrospray device by etching the features of the electrospray device from a monocrystalline silicon substrate without any need for assembly. The alignment scheme allows for nozzle walls of less than 2 μm and nozzle outer diameters down to 5 μm to be fabricated reproducibly. Further, the lateral extent and shape of the recessed annular region can be controlled independently of its depth. The depth of the recessed annular region also determines the nozzle height and is determined by the extent of etch on the nozzle side of the substrate.

[0192] An advantage of the fabrication process described herein is that the process simplifies the alignment of the through-wafer channels and the recessed annular region. This allows the fabrication of smaller nozzles with greater ease without any complex alignment of masks. Dimensions of the through channel, such as the aspect ratio (i.e. depth to width), can be reliably and reproducibly limited and controlled.

[0193] **FIGS. 19A and 19B** show a perspective view of scanning electron micrograph images of a multi-nozzle device fabricated in accordance with the present invention. The nozzles have a 20 μm outer diameter and an 8 μm inner diameter. The pitch, which is the nozzle center to nozzle center spacing of the nozzles is 50 μm.

[0194] The above described fabrication sequences for the electrospray device can be easily adapted to and are applicable for the simultaneous fabrication of a single monolithic system comprising multiple electrospray devices including multiple channels and/or multiple ejection nozzles embodied in a single monolithic substrate. Further, the processing steps may be modified to fabricate similar or different electrospray devices merely by, for example, modifying the layout design and/or by changing the polarity of the photomask and utilizing negative-working photoresist rather than utilizing positive-working photoresist.

[0195] Interface of a Multi-System Chip to a Mass Spectrometer

[0196] Arrays of separation-electrospray nozzles on a multi-system chip may be interfaced with a sampling orifice of a mass spectrometer by positioning the nozzles near the sampling orifice. The tight configuration of electrospray nozzles allows the positioning thereof in close proximity to the sampling orifice of a mass spectrometer.

[0197] A multi-system chip may be manipulated relative to the ion sampling orifice to position one or more of the nozzles for electrospray near the sampling orifice. Appropriate voltage(s) may then be applied to the one or more of the nozzles for electrospray.

[0198] Separation Block

[0199] The present invention also relates to a stackable separation block for use in effecting chromatographic separation of multiple samples. The separation block or stack of multiple separation blocks can be used alone or in combination with an electrospray device. The separation block can be made from any suitable material, preferably by conventional injection molding techniques. Each separation block has an array of a plurality of separation through-substrate channels. The multiple separation channels can be provided in an array having a spacing between adjacent channels and arrays having a density of channels corresponding to that of the entrance orifices of the arrays of multiple electrospray devices noted above. The separation material can be any material suitable for effecting a chromatographic separation of analytes, including polymer monolith, non-monolith polymer particles, particles containing a stationary phase, silica particles, non-porous silica, silica particles encapsulated in a polymer matrix, and the like. Stationary phase particles typically include from about 1 to about 60 μm, more preferably from about 1 to about 5 μm, most preferably about 1 to about 3 μm particle diameters coated with a stationary phase material. Separation particles can be retained in the channels by use of frits. The polymer monolith material can be fabricated in situ in accordance with the techniques noted above. Preferably, each channel of a particular block is filled with the same separation material or material having the same separation characteristics. The separation blocks can be used for sample preparation, as well as sample separation with or without integration with a microchip electrospray device.