

US007935924B2

# (12) United States Patent

Cheung et al.

# (10) **Patent No.:**

US 7,935,924 B2

(45) **Date of Patent:** 

May 3, 2011

## (54) BATCH FABRICATED RECTANGULAR ROD, PLANAR MEMS QUADRUPOLE WITH ION OPTICS

(75) Inventors: Kerry Cheung, Cambridge, MA (US);

Luis F. Velásquez-García, Boston, MA (US); Akintunde I. Akinwande,

Newton, MA (US)

(73) Assignee: Massachusetts Institute of Technology,

Cambridge, MA (US)

(\*) Notice: Subject to any disclaimer, the term of this

patent is extended or adjusted under 35

U.S.C. 154(b) by 157 days.

(21) Appl. No.: 12/168,439

(22) Filed: Jul. 7, 2008

(65) Prior Publication Data

US 2009/0026367 A1 Jan. 29, 2009

## Related U.S. Application Data

- (60) Provisional application No. 60/948,221, filed on Jul. 6, 2007.
- (51) **Int. Cl.** *H01J 37/12* (2006.01)
- (52) **U.S. Cl.** ...... **250/292**; 250/396 R

See application file for complete search history.

# (56) References Cited

### U.S. PATENT DOCUMENTS

3,553,451 A	* 1/1971	Uthe 250/290
5,726,448 A	3/1998	Smith et al.
6,403,955 B1	6/2002	Senko
6,441,370 B1	8/2002	Khosla et al.
6,465,792 B1	10/2002	Baptist
6,483,109 B1	* 11/2002	Reinhold et al 250/292
6,784,424 B1	8/2004	Willoughby et al.
6,797,950 B2	9/2004	Schwartz et al.
6,870,158 B1	3/2005	Blain
6,891,157 B2	5/2005	Bateman et al.
7,126,116 B2	2 * 10/2006	Ueno 250/290
7,208,729 B2	2 * 4/2007	Syms 250/288
7,329,879 B2	2* 2/2008	Kernan et al 250/396 R
7,457,708 B2	2 * 11/2008	Thompson et al 702/19
2009/0206250 A1	l * 8/2009	Wollnik 250/290

### FOREIGN PATENT DOCUMENTS

$\mathbf{SU}$	1396174	5/1988
SU	1758706	8/1992

<sup>\*</sup> cited by examiner

Primary Examiner — Kiet T Nguyen

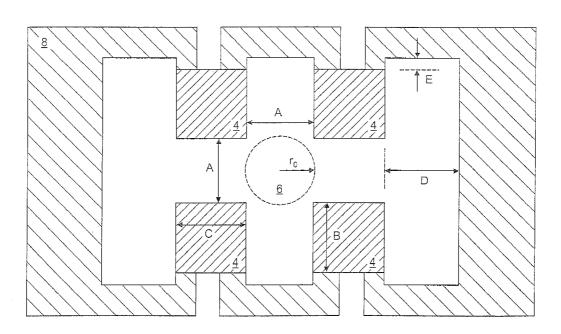
(74) Attorney, Agent, or Firm — Gauthier & Connors LLP

## (57) ABSTRACT

A quadrupole mass filter (QMF) is provided. The QMF includes a plurality of rectangular shaped electrodes aligned in a symmetric manner to generate a quadrupole field. An aperture region is positioned in a center region parallel to and adjacent to each of the rectangular shaped electrodes. An incoming ion stream enters the aperture region so as to be controlled by the quadrupole field.

## 21 Claims, 8 Drawing Sheets

~2



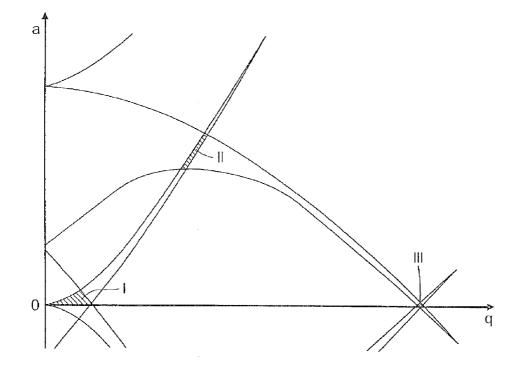
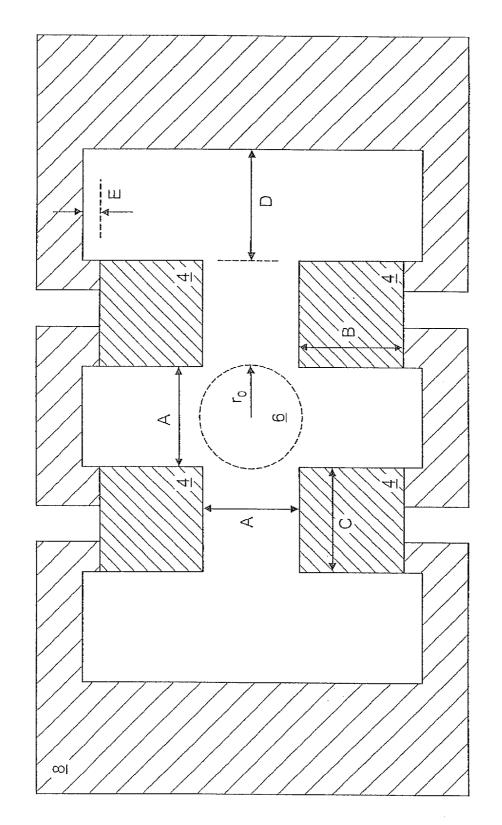
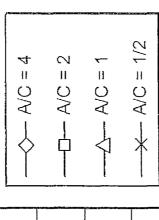
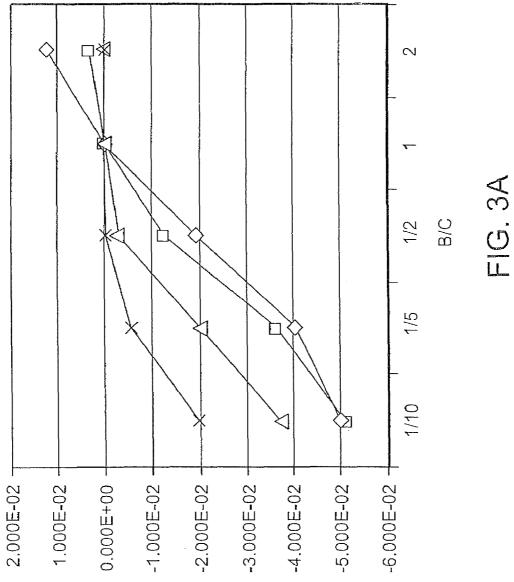


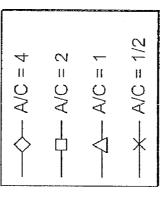
FIG. 1



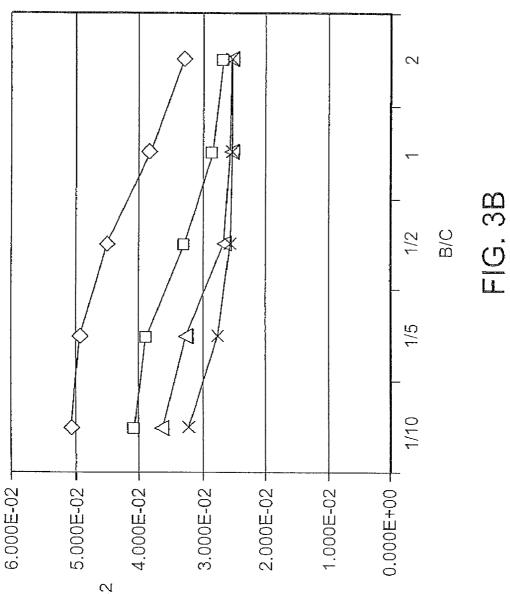


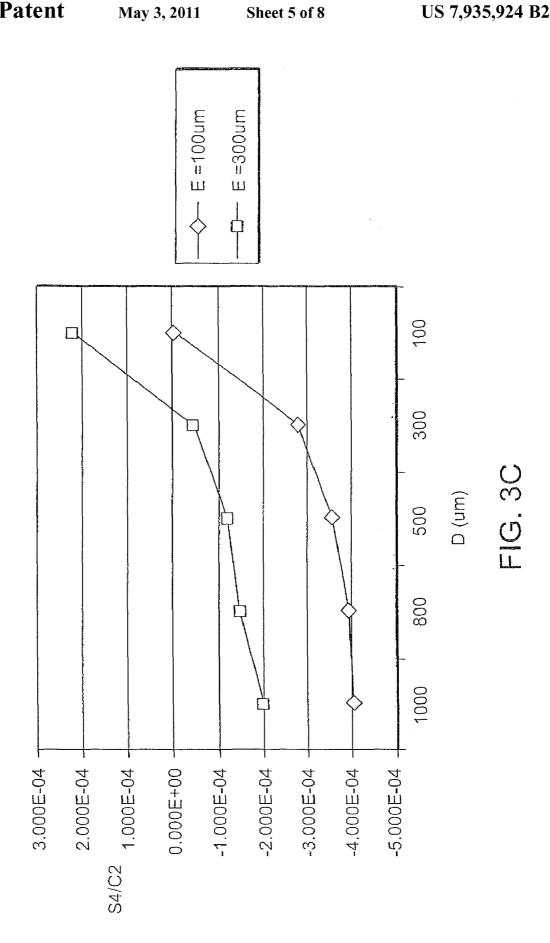
May 3, 2011

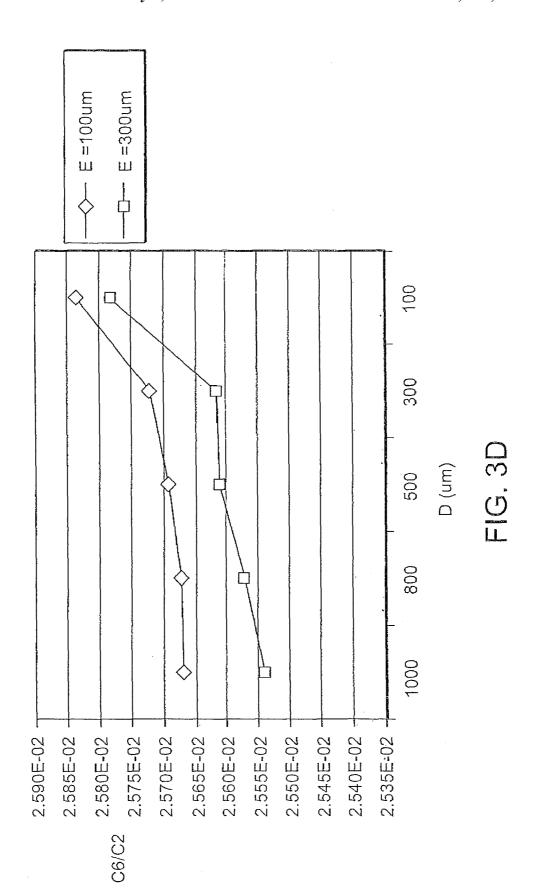


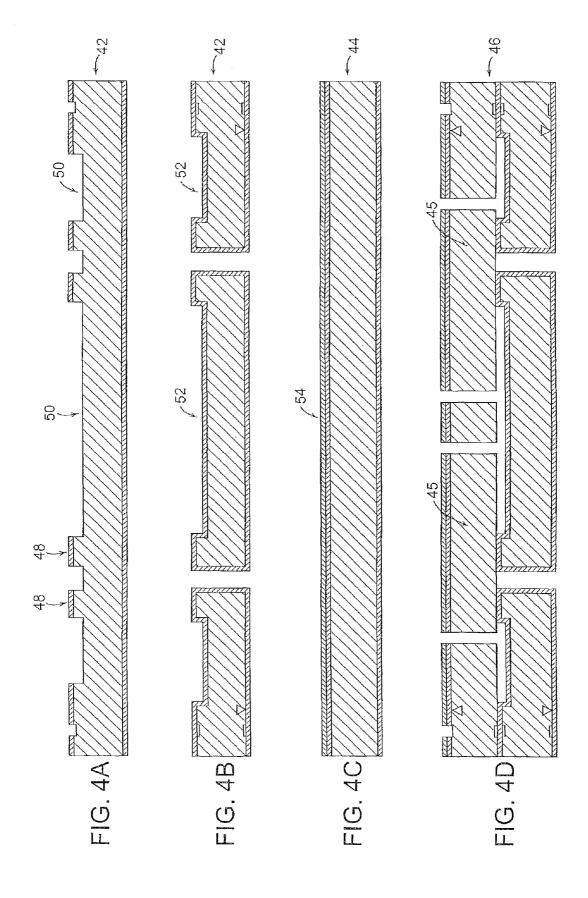


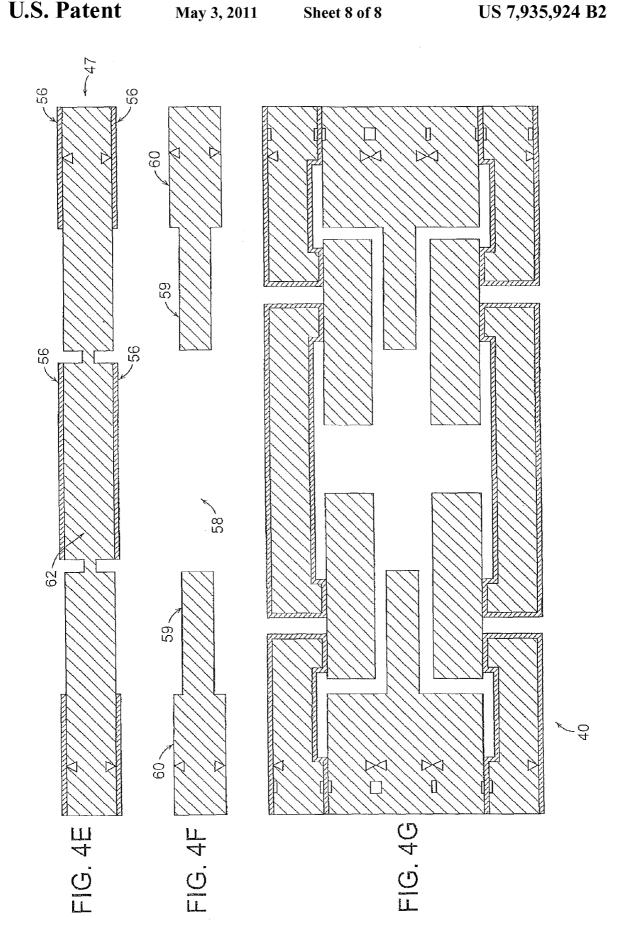
May 3, 2011











1

# BATCH FABRICATED RECTANGULAR ROD, PLANAR MEMS QUADRUPOLE WITH ION OPTICS

#### PRIORITY INFORMATION

This application claims priority from provisional application Ser. No. 60/948,221 filed Jul. 6, 2007, which is incorporated herein by reference in its entirety.

This invention was made with government support awarded by the Defense Advanced Research Projects Agency under Contract No. W911QY-05-1-000. The government has certain rights in the invention.

#### BACKGROUND OF THE INVENTION

The invention relates to the field of MEMS quadrupoles, and in particular to rectangular rod, planar MEMS quadrupoles with ion optics

In recent years, there has been a desire to scale down linear quadrupoles. The key advantages of this miniaturization are the portability it enables, and the reduction of pump-power needed due to the relaxation on operational pressure. Attempts at making linear quadrupoles on the micro-scale 25 were met with varying degrees of success. Producing these devices required some combination of microfabrication and/or precision machining, and tedious downstream assembly. For miniature quadrupole mass filters to be mass-produced cheaply and efficiently, manual assembly should be removed <sup>30</sup> from the process.

#### SUMMARY OF THE INVENTION

According to one aspect of the invention, there is provided a quadrupole mass filter (QMF). The QMF includes a plurality of rectangular shaped electrodes aligned in a symmetric manner to generate a quadrupole field. An aperture region is positioned in a center region parallel to and adjacent to each of the rectangular shaped electrodes. An incoming ion stream enters the aperture region so as to be controlled by the quadrupole field.

According to another aspect of the invention, there is provided a method of forming a quadrupole mass filter (QMF). 45 The method includes forming a plurality of rectangular shaped electrodes aligned in a symmetric manner to generate a quadrupole field. Also, the method includes forming an aperture region positioned in a center region parallel to and adjacent to each of the rectangular shaped electrodes. An 50 incoming ion stream enters the aperture region so as to be controlled by the quadrupole field.

According to another aspect of the invention, there is provided a method of forming a quadrupole field. The method includes aligning a plurality of rectangular shaped electrodes 55 in a symmetric manner to generate a quadrupole field. Also, the method includes positioning an aperture region in a center region parallel to and adjacent to each of the rectangular shaped electrodes. An incoming ion stream enters the aperture region so as to be controlled by the quadrupole field.

#### BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a Mathieu stability diagram showing quadrupole stability regions I, II, and III;

FIG. 2 is a schematic diagram of the inventive quadrupole mass filter cross-section;

2

FIGS. **3**A-**3**D are graphs illustrating the expansion used to examine the magnitudes of the higher-order components as a function of device geometry; and

FIGS. 4A-4G is a process flowgraph illustrating the fabrication of the inventive quadrupole mass filter.

### DETAILED DESCRIPTION OF THE INVENTION

The invention involves a purely microfabricated quadrupole mass filter (QMF) comprising of a planar design and a
rectangular electrode geometry. Quadrupole resolution is
proportional to the square of the electrode length, thus favoring a planar design since electrodes can be made quite long.
Rectangular rods are considered since that is the most amenable geometric shaped for planar microfabrication. This
deviation from the conventional round rod geometry calls for
optimization and analysis.

The inventive QMF utilizes four rectangular electrodes aligned in a symmetric manner to generate a quadrupole field. If the applied potential is a combination of r.f. and d.c. voltages, the equations of motion for a charged ion in this field would be given by the Mathieu equation. This equation has stable and unstable solutions that can be mapped as a function of two parameters. Overlapping the Mathieu stability diagrams for the directions orthogonal to the quadrupole axis define stability regions, shaded areas in FIG. 1, where ion motion is stable in both directions.

Most commercial QMFs and reported MEMS-based versions utilize cylindrical electrodes instead of hyperbolic ones due to the reduced complexity in manufacturing. To compensate for the distortion that comes from using non-hyperbolic electrodes, optimization was conducted to minimize the higher-order field components that are a result of this non-ideality. Optimization can be conducted on the rectangular electrodes of the inventive QMF to minimize unwanted field components as well.

FIG. 2 shows the cross-section of an inventive quadrupole mass filter 2. The quadrupole mass filter 2 includes four rectangular electrodes 4, aperture 6, and a housing unit 8. The rectangular electrodes 4 are aligned in a symmetric manner to generate and a quadrupole field. The aperture 6 is positioned in a center region parallel to and adjacent to each of the rectangular shaped electrodes 4, and allows an incoming ion stream to pass so as to be controlled by the quadrupole field. The rectangular electrodes 4 have a height B and width C. The aperture 6 includes a circular region having a radius  $r_0$  that is adjacent to the electrodes. The rectangular electrodes 4 are separated by a distance A and distances from the rectangular electrode surfaces to the surrounding housing are D and E.

Maximum transmission through a QMF occurs when the incoming ions enter near the aperture 6 of the QMF 2. The inclusion of integrated ion optics can help focus the ion stream towards the aperture 6, as well as control the inlet and outlet conditions, thus improving overall performance.

Maxwell 2D is used to calculate the potentials for the various geometries. The field solutions are exported into a MATLAB script that decomposed the field into equivalent multipole terms.  $C_2$  is the coefficient corresponding to an ideal quadrupole field, while  $S_4$  and  $C_6$  are the first odd and even higher-order component respectively. This expansion is used to examine the magnitudes of the higher-order components as a function of device geometry and is summarized in FIG. 3.

In simulations that excluded the housing, it is found that the coefficients  $S_4$  and  $C_6$  are minimized when the dimensions of the rectangular electrode (B or C) is equal to or greater than the dimension of the aperture (A) as shown in FIGS. 3A-3B.

3

Choosing an optimized electrode geometry with A=B=C and including the housing, simulations show that the distances from the electrode surfaces to the surrounding housing (D and E) should be kept equal to minimize  $S_4$ , but at the expense of  $C_6$  as shown in FIGS. 3C-3D.  $C_6/C_2$  is a minimum when D is  $S_4$  large as shown in FIG. 3D.

For fabrication and testing considerations, dimension A was set to 1 mm and E to 100  $\mu m$ . A large device aperture will increase the signal strength of the transmitted ions, while a small electrode-to-housing distance will improve processing uniformity. Although these dimensions were chosen, dimension A, B and C can range from 50  $\mu m$  to 5 mm while dimension D and E can range from 5  $\mu m$  to 5 mm or larger.

Higher-order field contributions arising from geometric non-idealities lead to non-linear resonances. These resonances manifest as peak splitting that is typically observed in quadrupole mass spectra. Reported work involving linear quadrupoles operated in the second stability region show improved peak shape without these splits. It is believed that operating the device in the second stability region will provide a means to overcome the non-linear resonances introduced by the square electrode geometry.

FIGS. 4A-4G are schematic diagrams illustrating the process flow used in describing the fabrication of the inventive quadrupole mass filter 40. Five highly-doped silicon double-side polished (DSP) wafers are needed to complete the inventive filter device. Two  $500\pm 5~\mu m$  wafers are used as the capping layers 42, two  $1000\pm 10~\mu m$  wafers serve as the rectangular electrode layers 44, and another  $1000\pm 10~g/m$  is utilized as a spacer layer 47. All the wafers initially have an oxide layer having a thickness of  $0.3~\mu m$  to serve as a protective layer 48 during processing.

A series of deep reactive ion etches (DRIE), wet thermal oxidation, and silicon fusion bonding is used to realize the device. Each of the cap wafers 42 is defined with release trenches 50 100 µm deep that are required for the electrode etch as shown in FIG. 4A, and through-wafer vias for electrical contact. The cap wafers 42 then have 1 µm of thermal 40 oxide 52 grown to serve as an electrical isolation barrier, as show in FIG. 4B. The electrode wafers 44 have 250 nm of silicon rich nitride 54 deposited on one side to serve as an oxide wet-etch barrier as shown as in FIG. 4C. The exposed oxide is removed with a buffered oxide etch (BOE) before 45 bonding to the cap wafers 42 and annealing. The electrodes 45 are defined in the bonded stack 46 with a DRIE halo-etch, as shown in FIG. 4D, followed by nitride removal with hot phosphoric acid. The spacer wafers 47 are coated on both sides with 4 µm of plasma enhanced chemical vapor depos- 50 ited (PECVD) silicon oxide 56 to serve as hard masks for a nested etch 62. On both sides, the PECVD oxide 56 is patterned with reactive ion etching (RIE), followed by DRIE of 450 μm to begin defining the aperture **58** as shown in FIG. **4**E. The entire spacer wafer 47 is then etched 100 µm on each side, 55 followed by an oxide strip 60 as shown in FIG. 4F. The nested etch 62 completes the aperture 58 and defines recesses 59 in the spacer wafer 47 which prevents electrical shorting in the final device. The thin protective oxide 48 on the cap-electrode stacks 46 are removed with BOE. The two stacks 46 and the spacer wafer 47 are then cleaned and fusion bonded, followed by die-sawing to complete the device 40 as shown in FIG. 4G.

The invention provides a fully microfabricated, mass-producible, MEMS linear quadrupole mass filter. A MEMS quadrupole with square electrodes can function as a mass filter 65 without significant degradation in performance if driving in higher stability regions is possible. Successful implementa-

4

tion of such devices will lead into arrayed configurations for parallel analysis, and aligned quadrupoles operated in tandem for enhanced resolution.

Although the present invention has been shown and described with respect to several preferred embodiments thereof, various changes, omissions and additions to the form and detail thereof, may be made therein, without departing from the spirit and scope of the invention.

What is claimed is:

- 1. A quadrupole mass filter (QMF) comprising:
- a plurality of rectangular shaped electrodes aligned in a symmetric manner to generate a quadrupole field;
- an aperture region positioned in a center region parallel to and adjacent to each of said rectangular shaped electrodes, an incoming ion stream enters said aperture region so as to be controlled by said quadrupole field; and
- a housing unit having a hollow rectangular cross-section that encloses said QMF, the inner surfaces of the housing unit being parallel to said rectangular shaped electrodes.
- 2. The QMF of claim 1, wherein said rectangular shaped electrodes are used for the purpose of ion optics, including but not limited to lenses, pre-filters, and post-filters, to improve device performance.
- 3. The QMF of claim 1, wherein the parameters of said rectangular shaped electrodes are optimized.
- **4**. The QMF of claim **1**, wherein the dimensions of said rectangular shaped electrodes are equal minimizes the first odd and even high-order components.
- 5. The QMF of claim 1, wherein the vertical and lateral distances between said rectangular shaped electrodes and said housing unit are equal so as to minimize high-order 35 components.
  - **6**. The QMF of claim **1**, wherein said rectangular electrodes have a separation distance between 50 µm and 5 mm.
  - 7. The QMF of claim 1, wherein the distances between said rectangular shaped electrodes and said housing are between 5 µm and 5 mm or larger.
  - **8**. A method of forming a quadrupole mass filter (QMF) comprising:
    - forming a plurality of rectangular shaped electrodes aligned in a symmetric manner to generate a quadrupole field: and
    - forming an aperture region positioned in a center region parallel to and adjacent to each of said rectangular shaped electrodes, an incoming ion stream enters said aperture region so as to be controlled by said quadrupole field; and
    - forming a housing unit having a hollow rectangular crosssection that encloses said QMF, the inner surfaces of the housing unit being parallel to said rectangular shaped electrodes.
  - 9. The method of claim 8, wherein said rectangular shaped electrodes are used for the purpose of ion optics, including but not limited to lenses, pre-filters, and post-filters, to improve device performance.
  - 10. The method of claim 8, wherein the parameters of said rectangular shaped electrodes are optimized.
  - 11. The method of claim 8, wherein the dimensions of said rectangular shaped electrodes are equal minimizes the first odd and even high-order components.
  - 12. The method of claim 8, wherein the vertical and lateral distances between said rectangular shaped electrodes and said housing unit are equal so as to minimize high-order components.

5

- 13. The method of claim 8, wherein said rectangular shaped electrodes have a separation distance of between 50  $\mu$ m and 5 mm.
- 14. The method of claim 8, wherein the distances between said rectangular shaped electrodes and said housing are 5 between 5 µm and 5 mm or larger.
  - 15. A method of producing a quadrupole field comprising: aligning a plurality of rectangular shaped electrodes in a symmetric manner to generate a quadrupole field;
  - positioning an aperture region in a center region parallel to and adjacent to each of said rectangular shaped electrodes, an incoming ion stream enters said aperture region so as to be controlled by said quadrupole field; and
  - enclosing said QMF with a housing unit having a hollow rectangular cross-section, the inner surfaces of the housing unit being parallel to said rectangular shaped electrodes.
- 16. The method of claim 15, wherein said rectangular shaped electrodes are used for the purpose of ion optics,

6

including but not limited to lenses, pre-filters, and post-filters, to improve device performance.

- 17. The method of claim 15, wherein the parameters of said rectangular shaped electrodes are optimized.
- **18**. The method of claim **15**, wherein the dimensions of said rectangular shaped electrodes are equal minimizes the first odd and even high-order components.
- 19. The method of claim 15, wherein the vertical and lateral distances between said rectangular shaped electrodes and said housing unit are equal so as to minimize high-order components.
- **20**. The method of claim **15**, wherein said rectangular shaped electrodes have a separation distance of between 50  $\mu$ m and 5 mm.
- 21. The method of claim 15, wherein the distances between said rectangular shaped electrodes and said housing are between 5  $\mu$ m and 5 mm or larger.

\* \* \* \* \*