Electrokinetic movement of the microparticulates between high resistance microelectrodes under the influence of dielectrophoretic force

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Abstract

Dielectrophoresis is a force applied to microparticles in non-uniform electric field. The presented study discusses the fabrication of the glassy carbon interdigitated microelectrode arrays using lithography process based on lithographic patterning and subsequent pyrolysis of negative SU-8 photoresist. Resulting high resistance electrodes would have the regions of high electric field at the ends of microarray as demonstrated by simulation. The study demonstrates that combining the AC applied bias with the DC offset allows the user to separate sub-populations of microparticulates and control the propulsion of microparticles to the high field areas such as the ends of the electrode array. The direction of the movement of the particles can be switched by changing the offset. The demonstrated novel integrated DEP separation and propulsion can be applied to various fields including in-vitro diagnostics as well as to microassembly technologies.

Keywords: Microassembly, Electrokinetic assembly, Microparticles, Dielectrophoresis, Carbon Electrodes

1. Introduction

Micro-assembly is an emerging tool for the assembly of micro-scale devices and parts with application to fabrication of sensors and surveillance devices, semiconductor devices, and Micro-Electro-Mechanical systems (MEMs) [1]. There are different types and forms of assemblies of micro-systems that have been studied. Serial assembly process can be exemplified by microrobots with vacuum grippers. Such robotic microassembly processes suffer from some technical drawbacks (such as microparticles plugging the vacuum pipes) and by time and expense of operating such systems [2]. Alternative micro-assembly approach that can be exercised on a massively parallel scale relates to fluid propulsion and guidance of the microparts. Such direct and indirect fluidic manipulation of particles can be driven by induced charge electro-kinetic phenomena, electrophoresis, electro-osmosis, dielectrophoresis, and electro-thermal flows [3]. Electro-kinetic assembly with dielectric forces can be used to move and place particles in specific locations. Using dielectrophoresis (DEP), transportation of micro-parts can be controlled by the geometry of the electrodes and channels, particle size, and material.

Dielectrophoresis is the movement of dielectric particles in a nonuniform electric field. It transports particles towards positions of the highest electric field gradient and can be either attractive – so-called positive DEP (pDEP) or repulsive – termed the negative DEP (nDEP). Whether the DEP force is positive or negative depends on the sign of Claussius-Mossotti factor K as shown in equation (1).

$$K = \frac{\epsilon_p^* - \epsilon_m^*}{\epsilon_p^* + 2\epsilon_m^*} \quad (1)$$

where ε_i^* stands for complex permittivity of the materials and subscripts p and m identify particles and suspension media, respectively [4].

Dielectrophoretic force, given by the equation (2), depends on the particle radius, *R*, the real part of the Claussius-Mossotti factor Re [K], and the gradient of the square of the electric field *E*:

$$F_{DEP} = 2\pi\epsilon_m R^3 Re[K](\nabla E^2) \quad (2)$$

Attractive and repulsive forces cause particles to be trapped in or move between the zones of high or low electric field. In an interdigitated electrode array, there are two sites having the strongest gradient of the electric field: the pits along the length of electrodes and the ends of the electrodes.

Present study looks at the propulsion of the polystyrene microbeads under the various electrical AC bias applied to the interdigitated electrode arrays (IDEAs) of carbon microelectrodes. DEP propulsion can provide a non-contact force under which particles can be directed toward the sites of strongest

electric field, and when coupled with pDEP or nDEP it can be utilized as a guided micro-assembly tool by non-contact forces for integrated separation, sorting, and agglomeration of microparts.

2. Materials and Methods

2.1. Fabrication

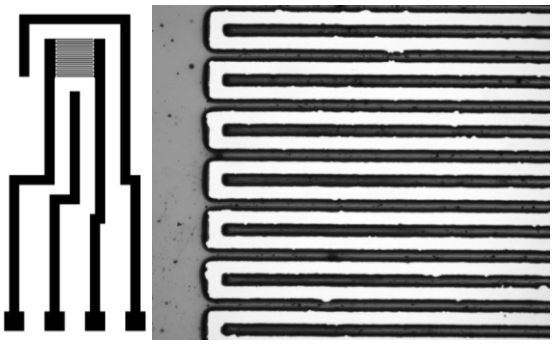

Fig. 1. (left) Schematic drawing of electrode with interdigitated design. (right) Micrograph of the interdigitated electrode arrays (IDEAs) used in the experiments. The width of the electrode is 40 microns.

For fabrication of interdigitated electrodes, a 100 mm diameter single-crystal Si (001) wafer with 10,000 A thick silicon dioxide was used as a substrate for fabrication of IDEAs. The electrode design schematics and the micrograph of the IDEAs used in this work is presented in Figure 1. The design consists of an array of fingers of width 40 μm, separation of 60 μm, and height of 2 μm.

A mask printed on an acrylic sheet (CAD/Art Services, Inc., OR, USA) was employed in the lithographic patterning of the negative resist for the production of the microelectrodes. Spincoater P6700 (Specialty Coating Systems, INC., IN, USA) was used to deposit SU-8 2025 negative photoresist (MicroChem, MA, USA) using sequence of spin at 500 rpm for 10 seconds followed by 2750 rpm for 30 seconds. The photoresist deposition was followed by soft bake on a Programmable Hotplate HP61 (Torrey Pines Scientific, CA, USA) by heat ramp of 120 ºC/hr from room temperature to 95 ºC with dwell at 95 ºC for 5 minutes, and natural cooling to room temperature. A Karl Suss MA6 Aligner (SUSS MicroTec, Garching, Germany), was utilized to expose the wafer with the resist to UV radiation with a dose of 100 mJ cm^{-2} for 60 seconds. After the UV exposure, the wafer with the exposed resist was baked again with the same temperature regime as that of soft bake. The wafer was then placed in a SU-8 developer for 5 minutes followed by the cleaning the wafer with acetone and isopropanol. Optical inspection confirmed the fidelity of the transferred pattern.

After the electrode design was produced in SU-8, the wafer was placed in the quartz tube of the furnace so that SU-8 IDEAs underwent pyrolysis to convert the organic resist into glassy carbon microelectrode arrays. After the purge with nitrogen at room temperature for 1 hour at 0.5 SCFH, the temperature was ramped at the rate of 25°/min and then the temperature was held at 300°C for 30 minutes. The temperature was further increased at the rate of 12°/min to 900°C and the held at 900°C for one hour followed by overnight passive cooling to room temperature. After the pyrolysis step the wafer was diced with a diamond point scribe into individual chips containing IDEAs.

2.2. Experimental Setup

The copper wires were soldered onto the carbon contact pads with Indium metal. The wires were then connected to the Function Generator DS345 (Stanford Research Systems, Sunnyvale, CA) to apply the desired AC frequency, Offset (V) and voltage (V). Solutions of Carboxylated polystyrene microbeads of 1 μm and 5 μm in diameter (Magsphere Inc., Pasadena, CA) suspended in the deionized water were pipetted onto the interdigitated electrodes and covered by the microscope glass slide to reduce evaporation and facilitate the observation of the movement of the beads. An Eclipse LV-UDM Universal Design Microscope (Nikon Instruments Inc., Melville, NY) was utilized to observe the electrokinetic phenomena.

3. Simulation

COMSOL Multiphysics 5.3a© (COMSOL Inc., Burlington, MA) platform was used to analyze the distribution of electric field that influences the movement of particles.

The electrode geometry studied in this work is a single SU-8 derived glassy carbon electrode pair that is a part of the interdigitated electrode array (IDEA). This single electrode finger has a width of 40 μm, a length of 2000 μm and a 60 μm edge-to-edge separation with adjacent electrodes.

The electric potential distribution represented in Figure 2 and the electric field distribution of Figure 3 was simulated for 10 V peak-to-peak bias with one electrode having 0V potential applied at one end of the finger and the other electrode had 10 V applied at the opposite end. The conductivity of the water solution is 0.05 $\mu S \cdot m^{-1}$ and the resistivity of SU-8 derived glassy carbon electrodes is $4 \times 10^{-5}\ \Omega \cdot m$. The relative electrical permittivity of glassy carbon electrodes and aqueous solution were 30 and 80.3, respectively.

The simulation demonstrates that the high field regions are located at the ends of the electrode arrays. These simulation results are consistent with

the experimental evidence discussed below that the beads would tend to move to these high field areas.

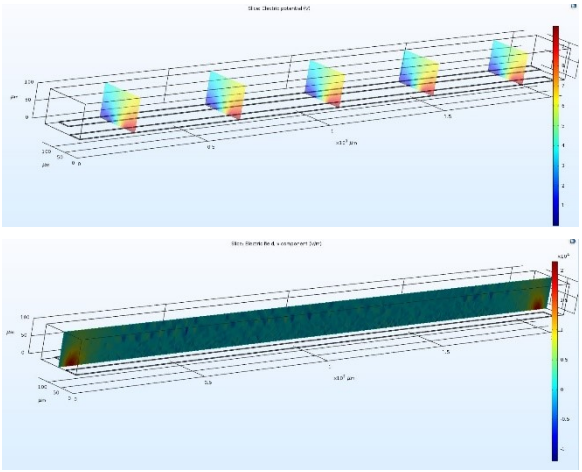

Fig. 2. COMSOL simulation of potential distribution(top) and electric field distribution (bottom) of the interdigitated electrode array.

4. Results and Discussion

The set of experiments was performed under constant amplitude and frequency with a varying offset to examine the phenomenon of DEP propulsion towards the ends of the microelectrode arrays. The offset represents a DC shift of the applied bias as represented in Figure 3. Introduction of the offset changes the physics of DEP forces significantly.

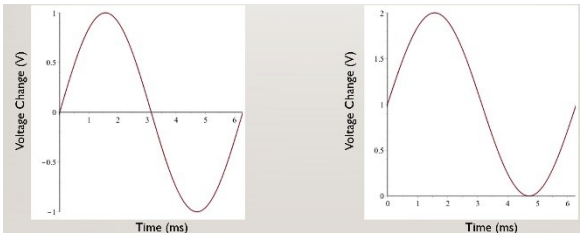

Fig. 3. Application of 2 Vpp with 0V offset (left) and with 1V offset (right).

When the offset is equal to zero, sinusoidal signal is symmetrical (half of the cycle is positive bias and half of the cycle – negative bias) and such AC DEP force is well described by the equation 2 above. When the CM factor is positive, the beads are attracted to the electrode fingers, typically, the increase in frequency will make CM factor negative for simple spherical beads, leading to negative DEP and repulsion of the beads from the electrodes [5]. The cross-over frequency of CM=0 was determined for 1 and 5 μm diameter beads to be 0.3-0.5 MHz and 0.4-0.5 MHz, respectively.

Therefore, for zero offset we observe AC DEP and, depending on frequency, we see either attracting of repulsion from neighboring electrode fingers as was reported earlier [5].

If a significant offset is applied, the bias is either mostly, or wholly positive or negative and thus we observe either wholly DC DEP or a mix of AC and DC DEP, depending on the value of the offset. It was discovered that the direction of the bead movement could be manipulated by change in the offset value.

While the details of the physical framework that underly mixed mode DC/AC DEP is beyond the scope of the present technical brief, results of the several experiments will serve to illustrate the range of possibilities of particle manipulation on the interdigitated carbon electrode microarray when frequency, bias, as well as the DC offset can be varied.

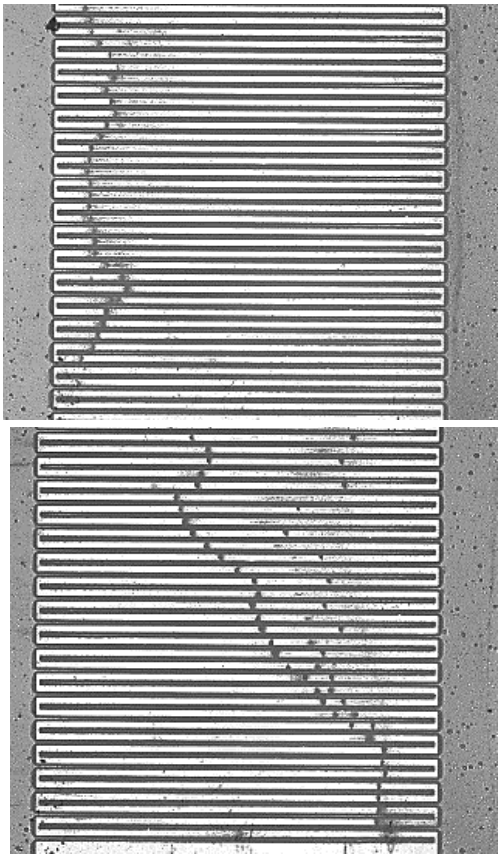

Fig. 4. 1 μm beads under the influence of 100 KHz,3 Vpp bias move to the left edge of the electrode array when the offset is + 1.3V (top), but movement of the beads switches to that towards the right edge when the offset is changed to -1.3Vpp (bottom).

For example, Figure 4 presents captured frames from the experiment where at the solution of 1 μm diameter beads under the amplitude of 3 Vpp and 100 kHz and the positive offset of +1.3 V can be seen moving to the left end of the electrode array. When the offset was switched to -1.3 V, the direction of movement changed and the beads started moving toward the right side under of the electrode array. Thus, a controlled propulsion and movement of the beads toward the regions of high electric field could be achieved successfully by offset manipulation.

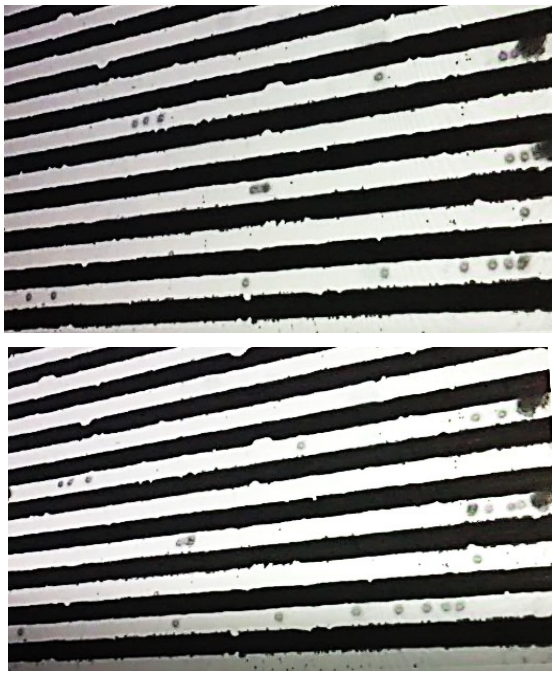

Fig. 5. The mix of 1 μm and 5 μm diameter beads is separated under the influence of 10 kHz, 3Vpp, 1V offset bias. The images (top and bottom) were captured 2 seconds apart. Smaller beads experience pDEP and are attracted to the electrode fingers (seen as small protrusions along the edges of the electrodes), while larger beads move to the left side of microelectrode array.

Additionally, DEP propulsion can be coupled with positive or negative DEP for the integrated separation of microparticles (such as different subpopulations of biological cells). For example, we have successfully achieved a separation of the mixed solution of 1 and 5 μm diameter beads into two groups of beads based on the respective diameter.

Figure 5 demonstrates sequence of frames captured 2 seconds apart where the mixed population of beads was separated since at 10 kHz, 3 Vpp the 1 μm diameter beads experienced positive DEP and were attracted to the electrodes, while 5 μm diameter beads experienced negative DEP and repelled from the electrodes (they can be seen between the electrodes). At the same time, 1 V offset was applied and 5 micron beads move to the left side of the electrode array. Subsequently, these beads could be collected and removed to a separate location if the goal of the process is the separation step only. However, the separation and propulsion combined can be utilized in microassembly to separate out one type of microcomponents and then propel them to a desired location, or in medical diagnostic platforms such as separating and analyzing circulating tumor cells.

5. Conclusions

A novel dielectrophoresis based propulsion technique utilizing high resistance electrodes has been discussed. The simulation describes field distribution within the system confirmed our results of particle moving toward high field at the end of the electrodes. The proof-of-principle operation of separation of 1 and 5 micron beads from the mix that integrated separation and propulsion steps has been demonstrated.

Acknowledgements

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