

Research in Microfluidic Transport at Sandia National Laboratories

The Microfluidics department of the Sandia National Laboratories is vigorously probing the chemical physics of microfluidic transport and developing high-performance, transport-based microfluidic systems. Sandia has made breakthroughs in electrokinetic pumping and in fundamental electroosmosis and dielectrophoresis. This fundamental and practical knowledge has assisted Sandia's development of μ ChemLab, a hand-held chemical analysis system. To support both basic and applied research, Sandia has developed innovative diagnostic instrumentation to characterize microflows and quantify microsystem performance and has validated numerical tools to simulate electrokinesis and dielectrophoresis.

Introduction

Recent engineering efforts to develop hand-portable liquid-phase analysis systems, such as μ ChemLab and μ ChemLab/CB, have motivated fundamental research into microfluidic physics and chemistry at Sandia National Laboratories. Recent work has analyzed fundamental electrokinesis (Cummings et al., 2000; Griffiths and Nilson, 2000a) and dielectrophoresis (Cummings and Singh, 2000). This research has produced geometrically optimized designs of low-dispersion turns for micro-analytical systems (Griffiths and Nilson, 2000b) and protocols for injecting samples in compact, controlled plugs for chip-based chemical separations. For example, true miniaturization of chromatographs has, until now, been impeded by the need to prevent dispersion in the column; simply "folding" a column into a compact space with U-turns leads to highly inefficient columns. Sandia's optimized low-dispersion turns make it possible to fold a 0.9-m chromatographic column into about 100 mm². The performance of many liquid chromatography techniques depends strongly on the shape and size of the sample plug

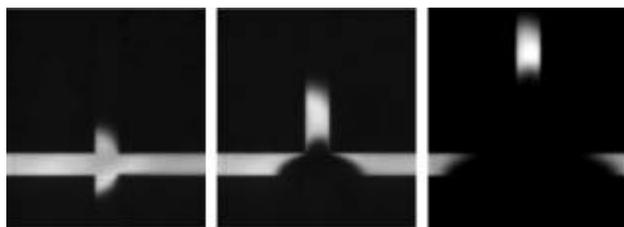


Fig. 1 Fluorescence images of an electrokinetic injection of a plug of fluorescent fluid sample. From a reservoir, the sample flows electrokinetically through the sample channel (horizontal) across the top of a chromatographic column (vertical). The applied fields are then adjusted to inject a well-defined, compact sample plug into the column, which is necessary for efficient analysis.

introduced into the column. Using numerical calculation, analysis, and quantitative visualization experiments, design rules have been developed for electrokinetic sample injection. An example of a "clean" sample injection is shown in Fig. 1. Work is continuing on this subject to treat complications encountered in practical systems, which often contain fluids with non-uniform properties. As a third example of recent work, we have developed a unique, experimentally validated capability for predicting electrokinetic and dielectrophoretic transport in arbitrarily complicated quasi-planar geometries germane to microsystems.

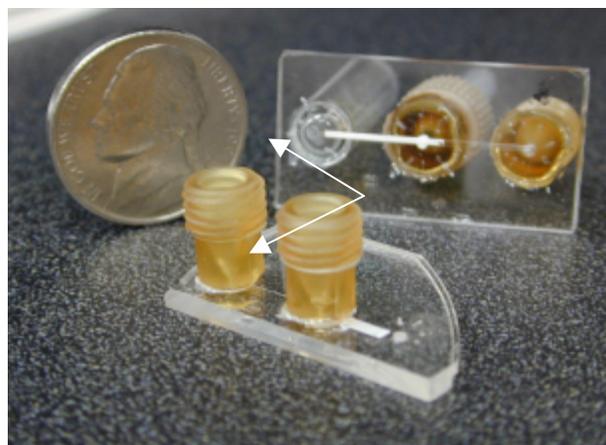


Fig. 2 Two glass chips with integrated electrokinetic pumps (EKPs) (thick white line in each chip; electrodes not shown). These EKPs produced a hydraulic pressure of over 1000 psi at their outlets.

Electrokinetic Pumping

Sandia National Laboratories has developed electrokinetic pumps (EKPs) that have demonstrated pressures up to 10,000 psi and flow rates over 100 μ L/min (Paul et al., 1998, 2000). Potential applications include replacement of HPLC pumps,

micro-valve actuation, and microprocessor cooling. EKPs can be fabricated in macroscale or microscale, depending on the flow rate required and the application. Figure 2 shows two microfluidic chips with integrated EKPs. In general, EKPs consists of a porous material and two electrodes submersed in the electrolyte at each end of the porous medium. An electric field applied across the porous medium generates electroosmotic flow (EOF). Pressure can be developed at the outlet until the pressure-driven back-flow through the porous medium matches the electroosmotic flow rate. The ratio of the maximum pressure to the applied voltage depends upon the working fluid and porous material properties. Up to 9 psi/Volt has been attained in our laboratory. In collaboration with two synthetic chemistry groups at Sandia, we are testing novel porous materials for electrokinetic pumps and other microfluidic systems. Diagnostics for the effective zeta potential, characteristic pore diameter, porosity, and other properties of interest have also been developed to quantify material performance.

Microflow Diagnostics

Sandia's micro-flow experimental capabilities are extensive and include accurate single-pixel-resolution velocity and velocity statistics measurement in transparent microchannels by the technique of particle-image velocimetry (Cummings, 2001). We have demonstrated <0.1% uncertainty and 0.8- μm -by-0.8- μm spatial resolution in velocity measurements over a field of view of ~ 0.5 mm by 0.4 mm. Figure 3 shows an overlay of experimentally measured and predicted speed contours of electrokinetic flow in uniform arrays of posts. This measurement technique can readily produce velocity measurements at better than 300-nm-by-300-nm spatial resolution for fundamental microscale fluid and particle mechanics studies. The imaging

technique is able to measure velocity histograms and correlations at this resolution, offering a new window into stochastic or chaotic microscale flow behavior. We have also developed an exciplex-fluorescence-based thermal-imaging capability for high-resolution thermometry in microchannels.

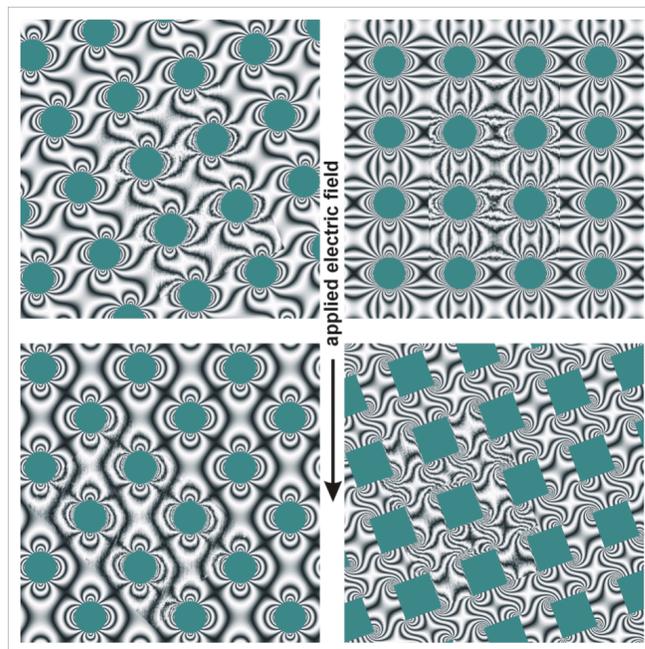


Fig. 3 Overlay of calculated and experimental electrokinetic speed-contour fields within uniform arrays of insulating posts on 200- μm centers. The contour spacing is 24 $\mu\text{m}/\text{s}$. The measurement spatial resolution is 0.8 mm. The mean flow is from top to bottom.

Another microflow imaging technique pioneered at Sandia is caged dye imaging. A caged dye is a fluorescent dye molecule and quenching moiety that are linked by a bond that can be cleaved by UV photons. When the bond is broken, the fluorescence of the dye molecule increases dramatically. Fluid

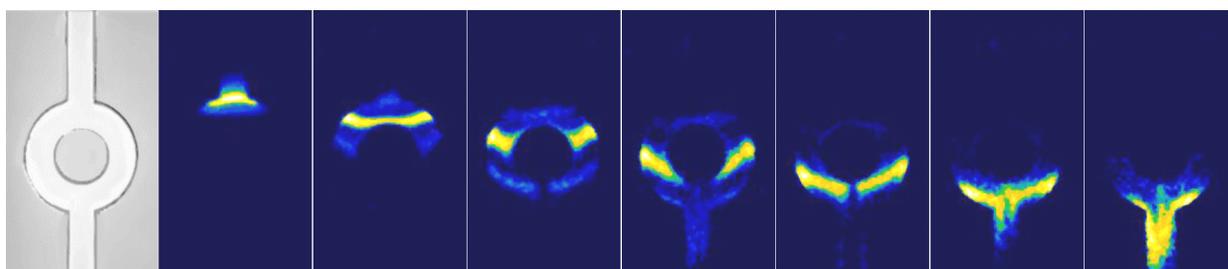


Fig. 4 Frames of a video showing an electrokinetic flow driven by ~ 40 V/mm in a microfluidic channel (50 mm wide x 10 mm deep) having a circular feature. A 10-mm-wide laser beam uncages dye molecules just above the circular feature. The fluid is 50:50 acetonitrile/phosphate buffer at pH 7.6. The faint leading blue band visible in the early frames is a positively-charged impurity which electrophoretically separates from the zwitterionic dye (yellow band).

motion may be visualized by uncaging dye in a fluid using light from a pulsed UV laser in a region in the flow, and observing the motion of the fluorescent region, as shown in Fig. 4. The method is useful for troubleshooting micro-Total Analysis Systems (μ TAS), because it reveals dispersion in a microfluidic system far more quickly and conclusively than traditional dye imaging techniques. For example, the image sequence in Fig. 4 shows the dispersion of a dye band during electro-osmotic flow around a circular obstruction. The faint blue band that leads the bright yellow band is a positive-charged impurity in the dye, which separates from the zwitterionic dye (yellow band) electrophoretically. We have also developed a variation of this imaging technique that employs photo-bleaching of a zwitterionic dye rather than uncaging. The depleted region of this dye convects without appreciable electrophoresis, simplifying the interpretation of the flow.

Dielectrophoresis

With our quantitative fluorescence imaging capability we have studied and developed predictive tools for mixed electrokinetic and dielectrophoretic particle flows in arbitrary planar geometries. Figures 5 and 6 show dielectrophoretic trapping and filamenting of 200-nm fluorescent particles in uniform arrays. The field at right in Fig. 5 is a contour map of the combined electrokinetic and dielectrophoretic trapping potential. The regions where these contours curve inward on the posts correspond to particle traps. Calculated flow streamlines are superimposed on the particle fluorescence image in Fig. 6. These mixed flows enable a variety of novel microfluidic devices.

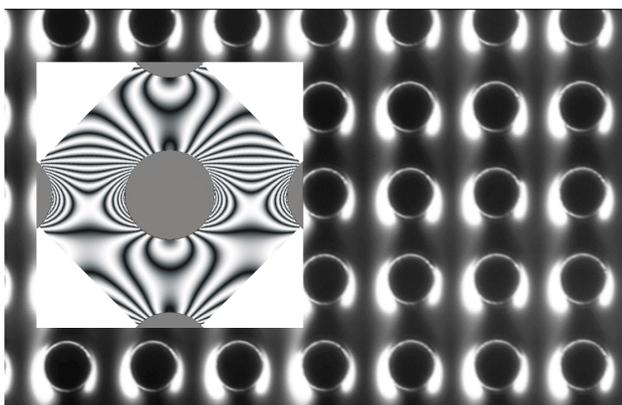


Fig. 5 Fluorescence image of particle trapping in an array via dielectrophoresis in a combined electrokinetic

and dielectrophoretic flow. The inset shows contours of the potential field experienced by particles. Trapping occurs where these contours begin and end on the same boundary.

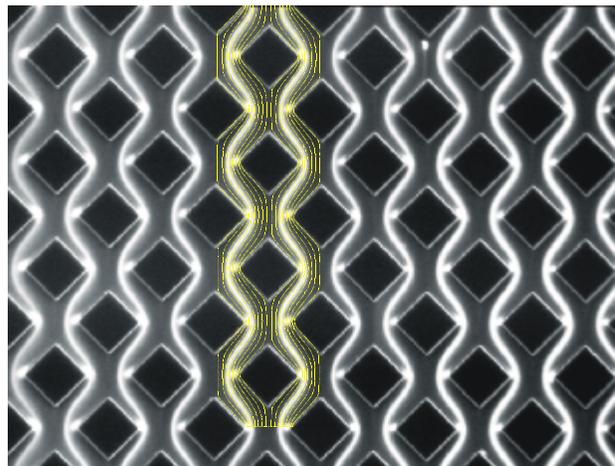


Fig. 6 Fluorescence image of filamentary particle flow produced by combined electrokinesis and dielectrophoresis. The flow is from top to bottom. Calculated streamlines are superimposed.

Microfabrication

Sandia's fabrication capabilities are extensive. These include the ability to design and fabricate complicated flow channels with embedded electrodes in glass, to pattern monolithic porous polymers and post arrays within channels, and to employ deep reactive-ion etching, LIGA, embossing, casting, and micro-injection molding to produce a microsystem with desired properties. Surface properties can be tailored using plasma oxidation. Sandia is exploring the use of silicon-based CMOS-compatible microfluidic systems for high-voltage electrophoresis and other applications.

References

- Cummings, E. B. (2001) An image processing and optimal nonlinear filtering methodology for PIV of microflows, *Exp. Fluids*, to be published in January 2001.
- Cummings, E. B., Griffiths, S. K., Nilson, R. H., Paul, P. H. (2000) Conditions for similitude between the fluid velocity and electric field in electroosmotic flow, *Anal. Chem.*, **72**, pp 2526–2532.
- Cummings, E. B., Singh, A. K. (2000) Dielectrophoretic trapping without embedded electrodes, Proc. of the SPIE Symposium on Micromachining and Microfabrication, Santa Clara, CA, September 18–20, *Microfluidic Devices and Systems III*, **4177**, No. 29, pp 164–173.
- Griffiths, S. K., Nilson, R. H. (2000a) Electroosmotic fluid motion and late time solute transport for large zeta potentials, *Anal. Chem.*, **72**, pp 4767–4777.
- Griffiths, S. K., Nilson, R. H. (2000b) Dispersion in Turns for Species Transport by Electrophoresis and Electroosmotic Flow, Proc. of the SPIE Symposium on Micromachining and Microfabrication, Santa Clara, CA, September 18–20, *Microfluidic Devices and Systems III*, **4177**, No. 29, pp 229–240.
- Paul, P. H., Garguilo, M. G., and Rakestraw, D. J. (1998) Imaging of Pressure- and Electrokinetically Driven Flows through Open Capillaries, *Anal. Chem.* **70**, pp 2459–2467.
- Paul, P. H., Arnold, D. W., Neyer, D. W., and Smith, K. B. (2000) Electrokinetic Pump Application in Micro-Total Analysis Systems: Mechanical Actuation to HPLC, in *Micro Total Analysis Systems 2000*, Kluwer Academic, The Netherlands, pp 583–590.
- Paul, P. H., Arnold, D. W., and Rakestraw, D. J. (1998) Electrokinetic Generation of High Pressures Using Porous Microstructures, *Micro Total Analysis Systems*, Kluwer Academic, The Netherlands, pp 49–52.