APPLICATIONS OF X-RAY MICRO-IMAGING, VISUALIZATION AND MOTION ANALYSIS TECHNIQUES TO FLUIDIC MICROSYSTEMS

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ABSTRACT

This paper describes x-ray imaging, visualization and analysis technologies which are utilized in an investigation of the micro-structure and micro-motion in various fluidic microsystems. More specifically, we have imaged and visualized the 3-D internal microstructure of Redwood Microsystems gas and liquid microvalves using x-ray microtomography and a massively parallel supercomputer, the Princeton Engine. In other experiments, x-ray microradiography was used to image fluid transport through the valves as well as valve actuation. In addition, we have begun to develop a method to visualize and measure steady-state flow in microchannels through the use of emulsions. The motion of the fluid and detailed flow pattern are directly viewable by motion of the liquid droplets. Finally, we have employed image motion analysis, bolstered by physical and geometric constraints derived from fluid mechanics, to recover meaningful flow fields and velocity profiles in microchannel flow. We found that our measured velocity profiles in 1000, 840, 750 and 640 micron inner diameter capillary tubes are in reasonably good agreement with the theoretical average velocity profiles of fully developed circular pipe flow.

INTRODUCTION

Numerous fluidic microdevices, such as valves, pumps and flow sensors, are rapidly emerging for chemical analysis and medical applications. To better design and fabricate these microdevices with increased reliability, a knowledge of the fluid flow behavior in this micron sized regime is crucial. However, an accurate model of flow behavior in the micron sized channels of these devices does not exist.

Most experimental studies of fluid transport have been limited to measurements of flow rates and pressure variations in flow systems or 2-D examination techniques such as optical microscopy. For example, J.N. Zemel and coworkers [1-3] have investigated flow in channels etched in Si ranging in area from 80 to 7200 square microns. In these experiments, the flow rate and the pressure drop were measured while varying the inlet pressure with a regulating valve. In addition, an optical microscope was used for direct observation of the fluid through the upper face of the channel i.e., a Pyrex glass cover bonded to the Si.

Our initial investigations have focused on the direct examination of the 3-D internal microstructure and microdynamics in microsystems using a combination of Exxon's x-ray microtomography capabilities, and Sarnoff's computer vision, visualization and supercomputing technologies. We have imaged and visualized the fluid streaming microscopically through various fluidic microvalves at high resolution. In addition, we have begun to develop a technique to visualize and measure steady-state flow in various micron-sized capillary tubes. Finally, we have developed vision-based fluid flow recovery algorithms

by incorporating physical and geometrical constraints to retrieve information about the physical flow of fluids from corresponding video imagery.

METHODS

X-Ray Imaging of Microsystems

Exxon has established an x-ray microtomography/microradiography facility at the National Synchrotron Light Source (NSLS), Brookhaven National Laboratories. This facility produces 3-D images of the internal structures of millimeter sized samples with resolution approaching 1 micron [4,5]. The synchrotron provides an intense, monochromatic, highly collimated x-ray beam with energies ranging from 5-30 keV. High quality microradiographs are generated with short exposure times of < 1 sec. The samples are uniformly illuminated by the x-ray beam. The wavelength is chosen so that the samples absorb approximately 80% of the incident x-rays. Differences in the x-ray absorption of materials form the basis of the radiography experiments. The phosphor screen converts x-ray light to visible light output for the CCD imager. A high power optical microscope objective is used to magnify the radiographic image formed in the phosphor onto the CCD camera. The digital image data is transferred to and stored in memory of a Macintosh computer.

Microtomography- Imaging Micro-Structure: This technique provides non-destructive, 3-D static characterization of internal microstructure of microsystems at high resolution. A typical experiment involves the collection of over one thousand radiographs or projections as a function of angle. These projections are reconstructed by a Direct Fourier Inversion algorithm to form 3-D opacity maps. Exxon's current reconstruction algorithm depends upon the sample being entirely covered by the x-ray beam throughout a 360 degree rotation.

Microradiography- Imaging Micro-Motion: This technique complements microtomography in that it provides information about the dynamics of the displacement process. Digital subtraction microradiography produces 2-D projections of the 3-D fluid flow through the channels where the background structure of the sample has been digitally removed from the image. Fluids are typically injected into a micro device by a syringe pump operating at a constant flow rate. The experiment proceeds by taking "pictures" (radiographs) approximately every second until enough frames are collected to visualize the displacement. Several hundred, 512 x 512 frames are accumulated. The sequence of radiographs are processed and then animated to create a "flow video" of the fluid moving through the device.

Visualization

Since the above instrumentation generates megabytes of data every few seconds, new computational methods for displaying, manipulating and extracting information from volumetric data sets and radiographs are

needed. Sarnoff has developed the Princeton Engine (PE) [6], a massively parallel supercomputer. Volume rendering algorithms have been implemented on the PE which enable the truly interactive volume visualization of the large, 3-D data sets collected at Brookhaven. These 3-D images, are computed and displayed in real-time, i.e., at multiple frames/sec [7]. We have also employed Silicon Graphics (SGI) hardware and software to display, clarify and enhance the flow data. Sample applications include simple viewers to display image sequences and volume data sets as well as various image processing and physical modeling tools.

Motion Analysis

A large body of work in image processing and computer vision deals with the extraction of motion information from image sequences [8]. Motion algorithms allow for the automatic detection and tracking of specific features of interest as well as for the estimation of a flow field in a given image sequence. Interestingly, relatively little has been done to make use of relevant physical and geometric constraints in applying image motion algorithms to specific domains of interest (e.g., the recovery of physical fluid flow) [9]. In the following paragraphs, we discuss image motion analysis, and how it is utilized to recover velocity flow fields.

As a point of departure, we are concentrating on the recovery of 2-D flow from pairs of temporally related 2-D images. The recovery of flow is a non-trivial matter due to local ambiguities in the apparent displacement of image intensities and the effects of noise [10]. Our approach to overcoming these matters is to constrain the flow recovery process by making use of physical and geometric constraints that are derived from fluid mechanics.

Currently, we are making use of three physically and/or geometrically motivated constraints on the flow recovery process. The first constraint enforces local rigidity of the recovered flow (i.e., translation and rotation of infinitesimal material particles). This is done by minimizing the total temporal derivative of imaged intensity, with intensity taken as a measurement of density. The second constraint enforces local smoothness of the recovered flow. This is done by minimizing spatial gradients in the recovered flow. The smoothness constraint is particularly important in ameliorating the effects of imaging noise. The third constraint concerns boundary conditions on the recovered flow. In particular, we enforce the boundary condition that at the interface of the fluid and its containing channel there can be no flow component orthogonal to the channel wall. Following [11] we combine these constraints and appeal to the calculus of variations [12] for a solution.

Algorithmically, we have embodied this iterative solution in a Jacobi relaxation scheme [13] and embedded it in a coarse-to-fine pyramid estimation framework [14]. The pyramid-component allows initial estimates to be computed at low spatial resolution and used as initial conditions for further refinement at successively increased levels of resolution. Making use of coarse-to-fine refinement increases the range of motions that can be recovered even while leading to greater computational efficiency.

RESULTS

We have applied the above mentioned x-ray imaging technologies in an investigation of micro-structure and micro-motion in representative microsystems e.g. fluidic microvalves and micron sized capillary tubes. This technology was demonstrated using three example microsystems described below. Note: The volume data sets and dynamic image sequences are best illustrated on video tape.

Redwood's Normally Open (NO) Microvalve

The Fluistor microvalve, designed and fabricated by Redwood Microsystems [15], has been imaged using x-ray microtomography. A cavity, etched in a Si substrate, is filled with a control liquid. When the liquid is heated, the Si membrane flexes outward toward the valve seat. The valve contains channels and holes designed to regulate fluid transport. We have worked with several such valves including a Normally Open (NO) gas microvalve and a NO liquid microvalve. Figure 1 shows a snapshot of the 3-D microstructure of the NO gas microvalve. After rescaling the volume in terms of color and opacity, features of interest or selected materials can be identified inside the microdevice. For example, the silicon substrate and pyrex glass in Figure 1 were set to be transparent for easy presentation of the inner structures.

Figure 1. Snapshot of the 3-D microstructure of Redwood's NO Fluistor Microvalve. Spatial resolution is 6 microns/voxel.

The above microtomography experiments were coupled with a measurement of mechanical motion in this valve by using microradiography. For example, an image sequence of the NO gas microvalve opening and closing was collected. We have observed that when the power is switched on, the membrane quickly snaps outward towards the valve seat or orifice. When the power is switched off, the membrane slowly relaxes back to its original position. The boiling of the control liquid in the cavity is also apparent in the radiographs. A microradiograph of the valve in the closed position is shown in Figure 2.

Figure 2. Microradiograph depicting a closed Fluistor Microvalve. The spatial resolution is 6 microns/pixel.

Digital subtraction microradiography, as described above, was also utilized to directly examine fluid transport. For example, we have investigated the initial filling of

Redwood's Normally Open liquid microvalve with iododecane. Iododecane was injected into the valve at a constant flow rate of $0.12\mu\text{l}/\text{min}$. We have observed the liquid initially wetting the space (air gap) between the silicon membrane and the Pyrex. Only after complete filling of the air gap, does the fluid proceed through the output port. We can clearly see the onset of an air bubble forming. Three microradiographs selected from this image sequence are shown in Figure 3. The arrows indicate the direction of flow through the valve. Bubble formation and dead space were prevalent during these experiments.

Figure 3. Microradiographs depicting the initial filling of Microvalve (after digital subtraction of background structure). The arrows indicate the direction of flow into the input port, through the air gap and finally leaving the output port. The spatial resolution is 6 microns/pixel.

Redwood's Normally Closed (NC) Microvalve

Microradiographs of an open (hot) and a closed (cold) NC valve are shown in Figure 4a and 4b, respectively. In this valve, the orifice is off-center with respect to the cavity and the membrane includes a fulcrum point. As the liquid is heated, the membrane pushes outward, causing the upper pyrex to rotate away from the orifice, allowing flow to occur. We have collected microradiographs of the NC valve during actuation and under varying inlet pressures. We have observed that increasing inlet pressure causes the upper pyrex to rotate towards the closed position, even though the valve is completely open (hot).

Micro Pipe Flow

We have begun to develop a technique to provide the simultaneous visualization and measurement of velocity flow field in microsystems. This technique can be utilized for steady state and unsteady state flows at the micron level. To accomplish this, an x-ray detectable contrast medium is first mixed with a fluid, e.g., oil mixed with water. After emulsification, the contrast medium is dispersed in the fluid in the form of fine droplets (diameter $1\text{--}20\mu\text{m}$). Since the density of the contrast medium is very close to the density of water, millions of the fine droplets are neutrally buoyant and suspended in the flow field. The droplets follow the motion of the fluid in a micro channel and are directly viewable with x-ray imaging methods. Several image sequences were collected using this technique to measure steady state flow of a liquid in quartz capillary tubes of various sizes. In these particular experiments, fluid was injected at a constant flow rate from 0.004 to $0.008\text{ microliters}/\text{sec}$. The qualitative flow field can be visualized by the motion of the droplets. In addition, the fluid velocity at any point can also be derived from the droplet movement between successive images and the known time interval between the images.

One image from a microradiograph pair is shown in the left panel of Figure 5. This image corresponds to an 840

micron quartz capillary tube with an emulsion solution being driven through at $0.004\text{ microliters}/\text{sec}$ captured at 500 msec exposure time. Examination of the image pair by eye suggests that in the (projected) center of the tube the emulsion droplets are displacing by approximately 10 pixels with a smooth transition to almost zero displacement at the (projected) left and right boundaries of the tube. The overall direction of motion is approximately vertical, with somewhat more directional variation in the center of the tube than at the sides. The right panel of Figure 5 shows the flow field recovered with our variational flow recovery algorithm.

The experimental velocity profiles across the channel at different flow rates, 0.004 and $0.008\text{ }\mu\text{l}/\text{sec}$, are plotted in Figure 6. These profiles were compared with the theoretical average velocity profile of fully developed circular pipe flow at the same flow rates and show reasonable agreement[16]. The recovered velocity profiles in 1000, 840, 750 and 640 micron diameter capillary tubes were collapsed into a non dimensional velocity profile, as shown in Figure 7. The radius of the capillary tube, R , and the maximum velocity at centerline, U_{max} are used to non-dimensionalize the plot. A fully developed pipe flow velocity profile is also superimposed as a solid line in the same plot. We find that this theoretical profile fits the recovered velocity profiles very well. This means that the flow within our test range is still dominated by viscous and pressure forces.

Figure 4. Microradiographs depicting Normally Closed Gas Microvalve a) closed (cold) and b) open (hot). The spatial resolution is 6 microns/pixel.

CONCLUSIONS

We have developed technologies which have allowed us to image, visualize and analyze micro-structure and micro-motion in fluidic microsystems. We have imaged and visualized the 3-D micro-structure and 2-D micro-fluidic motion in representative microdevices such as Redwood Microsystems' Normally Opened and Normally Closed Gas

Microvalves as well as their new Liquid Microvalve using x-ray microtomography and a massively parallel supercomputer, the Princeton Engine. We found that this technology can be used to verify the functionality of a particular valve as well as detect problem areas such as air bubbles and dead space. Finally, we have developed a method to image and visualize steady state fluid transport in various micron-sized quartz capillary tubes using emulsions. We have measured velocity profiles in 1000, 840, 750 and 640 micron inner diameter capillary tubes and found that they are in reasonably good agreement with the theoretical average velocity profiles of fully developed circular pipe flow.

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Figure 5. The left panel shows one image from an input image pair. The right panel shows the flow field recovered by our variational flow recovery algorithm. The channel diameter is 840 microns. The spatial resolution is 3 microns/pixel.

Figure 6. Comparison of experimental velocity profiles with theoretical (solid line) fully developed pipe flow average velocity profile

Figure 7. Nondimensional average velocity profiles experimental versus theoretical (solid line)