Three Dimensional Imaging of Structure and Flow—Critical to Advances in Microfluidics

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Microfluidics, the study of fluid flow through structures with micrometer scale dimensions, is an increasingly important discipline within a number of commercial and industrial applications. One focus of active microfluidic research at the Stanford University Microscale Heat Transfer Laboratories (MHTL) is mass and heat transport in two-phase flows, which has applications in the cooling of integrated circuits and the management of water created in PEM fuel cells. At its core, two-phase microfluidics is the study of interactions between moving liquids and/or gases and/or solids (though not necessarily stationary) structures. Advanced confocal microscopy, with its ability to visualize and measure both flow and structure on a single instrumental platform, will certainly play a key role in the continuing development of microfluidic devices.

Advanced Confocal Microscopy

Advanced Confocal Microscopy (ACM) refers to the latest generation of high-speed confocal microscopes, such as the Hyphenated Systems 3D MAP currently installed at MHTL (Figure 1). Like conventional optical microscopy, it uses visible light and can provide sub-micrometer aerial resolution. As do all confocal microscopes, it provides sensitivity in the *Z*-direction by using an aperture located in the imaging plane to exclude light rays that originate above or below the object plane. Unlike scanning confocal techniques, which assemble an image from a two-dimensional array of measurements acquired point-by-point as a finely focused laser scans the object, ACM acquires image information simultaneously through multiple apertures arranged in a spiral pattern on a rapidly spinning disc. Though modern scanning techniques are relatively fast, the multiple apertures and parallel data collection give ACM a fundamental speed advantage in image acquisition.

Figure 1: 3D MAP Advanced Confocal Microscope (ACM) installed at Stanford University Microscale Heat Transfer Laboratories (MHTL).

Although disc-based confocal microscopes have been available for decades, the performance of earlier generations was severely limited by available computing power. With modern processors and programming techniques, ACM can acquire a two-dimensional slice through the sample in a small fraction of a second, and can assemble and render a high-resolution, three-dimensional model composed of hundreds of such slices in a few seconds.

Other aspects of the system at MHTL are valuable in microfluidic applications. The ability to selectively exclude signals from overlying transparent layers, such as the glass cover slips often used to enclose microfluidic systems, permits imaging of subsurface features. Open and flexible software architecture permits easy integration with experimental control protocols and ancillary data collection and analysis techniques. An inverted optical system permits observations from below the sample and facilitates access from above for other experimental apparatus.

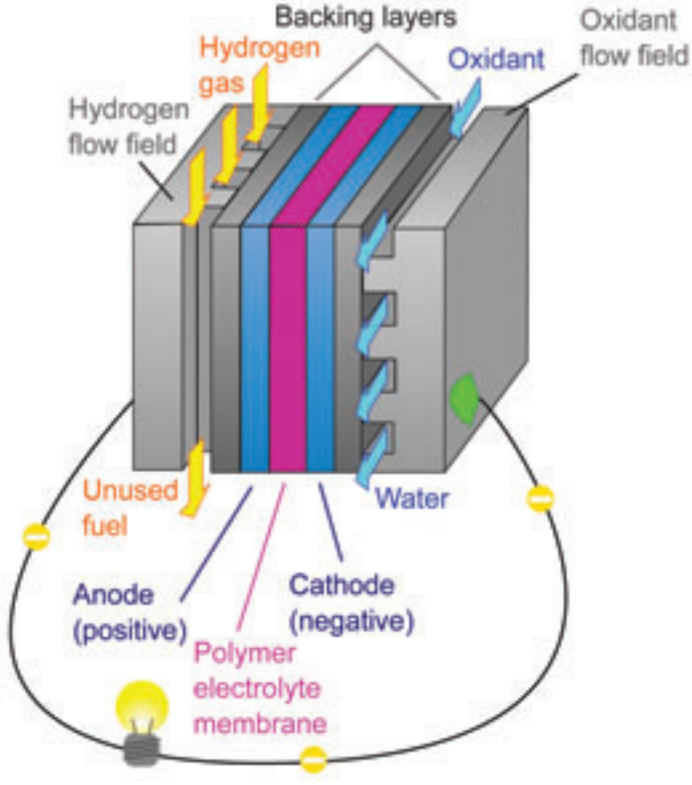

Figure 2: In proton exchange membrane fuel cells the membrane is permeable to protons but not hydrogen or oxygen. Electrons, separated from hydrogen at the anode by a catalyst, travel through an external electrical circuit before recombining with the protons and oxygen at the cathode. Water formed at the cathode must be transported out of the fuel cell.

Flow Characterization

A number of techniques are available for visualizing and quantifying microfluidic flow using various dyes, bleaches, and marking particles. Caged fluorescence uses a fluorescent dye

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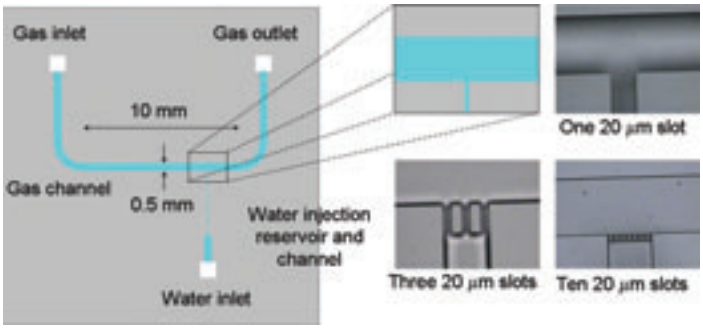

Figure 3 shows a test structure used to investigate two phase flows. Pressurized air flows through the u-shaped channel and fluid is introduced through the small channel perpendicular to it. The liquid port may contain a single or multiple slots to test various liquid injection conditions. The flow structure is mainly determined by the hydrophobicity of the channel walls. As illustrated in figure 4, hydrophilic walls lead to stratified flow conditions while hydrophobic walls tend to induce slug flow. In actual fuel cells channels the gas diffusion layer is usually hydrophobic and the channel walls hydrophilic, creating a mixed flow as shown in cross section at the bottom of the figure.

that is initially in a "caged" non-fluorescent state. After the dye is introduced into the experimental flow, a pulse of ultraviolet light breaks the bonds that cage the fluorescent group. The initial distribution of the uncaged fluorescent marker is thus confined to only the region illuminated by the activating light. At some time after activation, the now fluorescent dye is excited by a brief pulse of laser light, and its distribution captured by an imaging device. Bleached fluorescence is the converse of caged fluorescence. In bleached fluorescence techniques, the fluorescent dye is converted to a non-fluorescent state by a photo bleaching pulse from an argon laser.

Particle image velocimetry (PIV), and closely-related particle tracking velocimetry (PTV), are sophisticated image analysis techniques that characterize flows based on the movement of marker particles. Micro PIV is an adaptation designed for use with microscale flows. PIV cross-correlates sequential images of large numbers of marker particles to derive flow measurements. PTV actually tracks the movements of individual particles between images. PTV has the advantage of being able to separate the effects of random processes (such Brownian motion) and particle interactions from the larger scale flow.

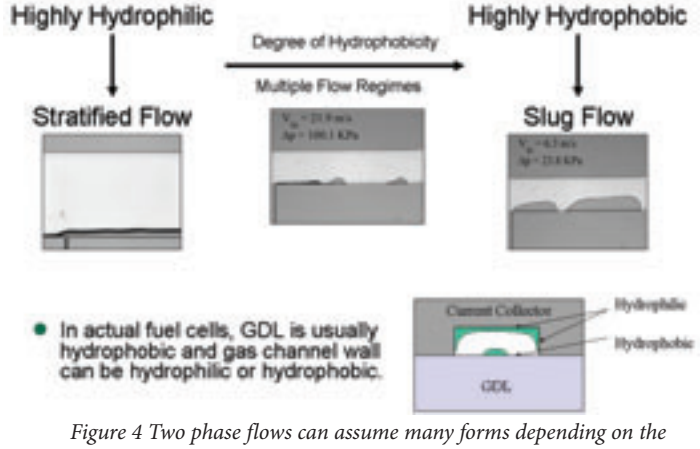

Figure 4 Two phase flows can assume many forms depending on the hydrophobicity of the channel material. In fuel cells the collector is often hydrophilic and the gas diffusion layer (GDL) hydrophobic, resulting in a mixed flow with stratified and slug components.

With ACM it should be possible to develop three-dimensional PIV (and PTV). Conventional particle image velocimetry techniques rely on the use of 2D images and are thus not capable of resolving the third spatial component of the velocity field. The three-dimensional imaging capability of ACM could enable three-dimensional particle image velocimetry, if sequential images can be acquired fast enough to render quasi-instantaneous volume distribution of particles that would allow valid cross correlation and tracking. Three dimensional particle velocimetry is an area of active research and development in a number of laboratories.

Microfluidic Flow

Fluid flow behavior in micro channels is quite different from that observed at the macroscopic scale in our everyday experience. Single phase microfluidic flows are typically laminar, lacking the turbulence and mixing that occurs readily in larger channels. This is due to the dramatic decrease in characteristic length (and therefore Reynolds number) that reduces the influence of inertial forces relative to viscous forces. Likewise, surface effects, such as surface tension, become predominant as the surface area of the channel walls grows in proportion to channel volume. In addition, when channel dimensions approach the mean free path between fluid molecules collisions and/or characteristic spacing, continuum mechanics and its assumptions break down. One such example is the no-slip boundary condition at the surface of the channel (that flow velocity is zero at the channel wall and increases smoothly with distance from the wall) no longer applies, and slip flow, a regime first explored in high speed aerodynamic analysis, develops. This is particularly true for gases, where the Knudsen number can be much smaller than one in microchannels. Clearly, things are quite different down there and successful development of microfluidic devices for commercial and industrial applications requires a thorough understanding of the physics that govern flow at the microscale.

The applications mentioned above, microelectronic cooling and fuel cell water management, are further complicated by the simultaneous presence of two phases, liquid and gas, in the flow. Two phase flows come in a variety of flavors defined in terms of their characteristics behavior and structure. Stratified flow occurs when the liquid forms a thin continuous layer, usually in contact

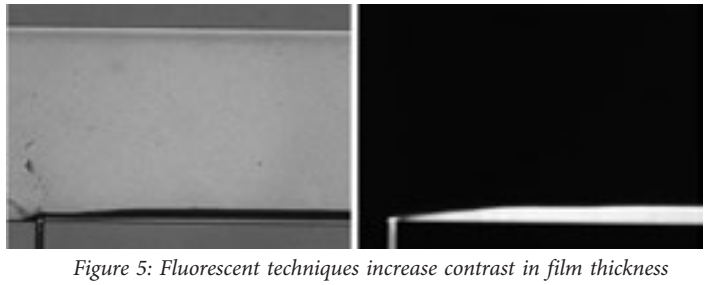

Figure 5: Fluorescent techniques increase contrast in film thickness measurements, but are only two dimensional.

with the channel walls. In slug flow, the liquid occurs in discontinuous droplets along the channel walls. In dispersed flow, small droplets of liquid are dispersed within the gas. Plug flow refers to alternating segments of gas and liquid that fully span the channel. And then there is mixed flow, which can comprise two or more of the other types. And then there is a whole set of descriptors that apply to flows that are predominately liquid with vapor bubbles

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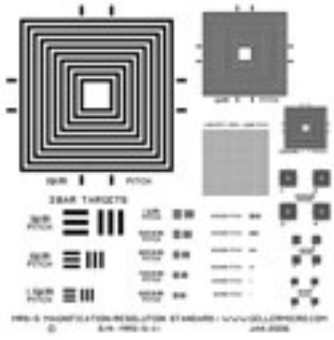

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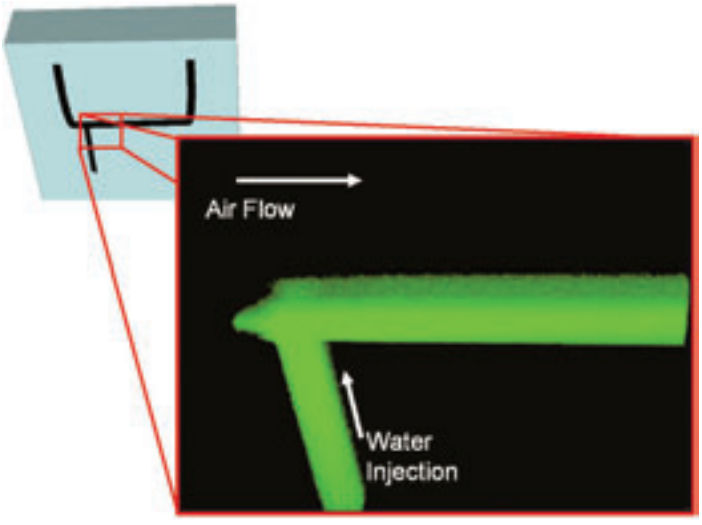

Figure 6 Three dimensional ACM measurements can measure film volume directly.

of various sizes and distributions.

Fuel Cell Applications

Most fuel cells today are of a type known as polymer electrolyte membrane (or proton exchange membrane) fuel cells—PEMFC (figure 2). As their name implies, they are distinguished by the use of a polymer membrane to mediate the proton exchange that occurs during their operation. The membrane is permeable to protons (H^+) but mostly impermeable to hydrogen and oxygen. PEMFCs are currently the best candidate for transportation and portable applications. They operate at relatively low temperatures (~80 °C), contain no caustic or toxic chemicals, and require only hydrogen and oxygen from air to operate, water being their only by-product.

In a PEMFC the membrane is sandwiched between porous, electrically conductive electrodes, typically a carbon fiber material. The membrane and electrodes are in turn sandwiched between conductive collector plates that contain channels to distribute hydrogen to the anode and to distribute oxygen and extract water and heat from the cathode. The electrochemical reactions that generate current occur on the surface of platinum catalyst particles that coat the carbon fibers of the electrodes at the interface with the membrane. At the anode, hydrogen splits into protons, which traverse the membrane, and electrons, which travel through an electrical circuit to the cathode. At the cathode the protons, electrons and oxygen combine to form water.

Water management is a significant challenge for PEMFC. The membrane requires a certain amount of water to remain permeable to protons. However, water created at the cathode must be removed since its excessive accumulation reduces the ability of gaseous oxygen to reach the catalyst particles, a condition known as cathode flooding. Capillary force is the predominant driving mechanism that moves water through the porous cathode and into the distribution channels of the collector plate. The focus of our research at the MHTL is the transport of liquid water, in a two phase flow propelled by the air supply, through these micro scale channels.

Figure 3: A silicon based microfabricated experimental structure was developed to investigate the pressure-driven, two-phase

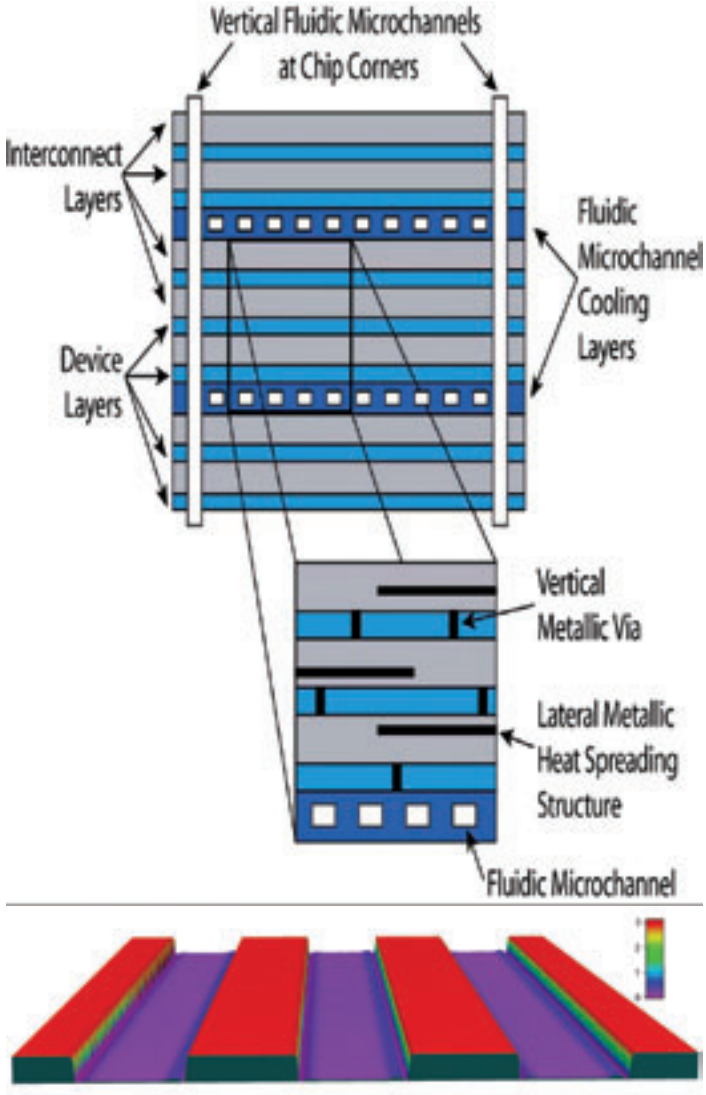

Figure 7 Top drawing -- Heat dissipation is a major challenge in the development of three dimensional integrated circuits (3DIC) that stack multiple dies. Microfluidic channels integrated into the interconnect architecture to circulate cooling fluids are a potential solution. Allowing the fluids to boil would increase the heat transport efficiency but requires a thorough understanding of two phase flows in microchannels. Bottom image – 3D MAP image of fluidic microchannels, each channel approximately 12 microns in width.

flow that removes water from the fuel cell (structures fabricated by Fu-Min Wang).

Fluorescence microscopy is a central tool for flow characterization. The sample fluid can be "stained" with a fluorescent dye or seeded with fluorescent, micrometer size (or smaller) particles, that emit light of a known wavelength when illuminated by light of another specified wavelength. Filters eliminate the illuminating and background light, making the fluorescence easy to distinguish within the image. Fig 5 compares a conventional white light image with a fluorescence image of a stratified flow. The enhanced image contrast provided by the fluorescence can be used for highly accurate quantitative film thickness measurements.

Although fluorescence improves the quality of such measurements, it is only two dimensional—it provides film thickness in the image plane only. Characteristics such as liquid volume would be better measured with a three dimensional techniques such a

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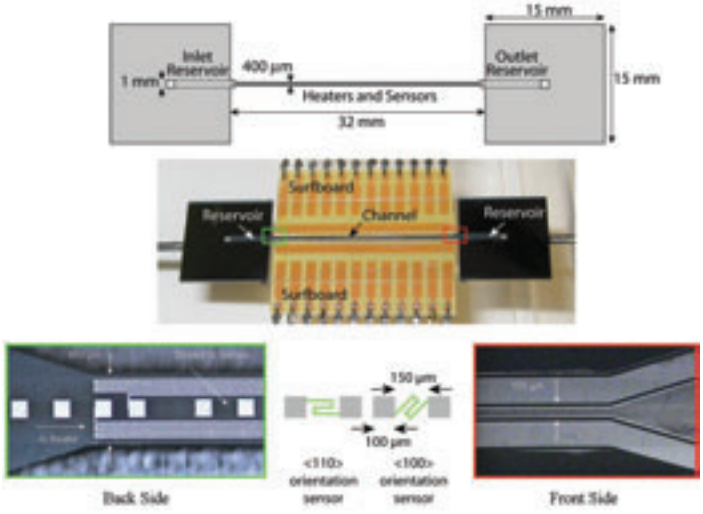

Figure 8 An isolated microchannel with integrated heating and temperature sensors is used to explore the behavior of boiling liquids in microchannels. (Images courtesy of Roger Flynn)

confocal microscopy (Fig 6).

Capillary forces play a predominant role in determining the behavior of two phase flows. Contact angle measurements that quantify surface tension can be difficult to acquire within a micro channel. The quantitative, three-dimensional imaging capabilities of ACM could permit fast and accurate measurements of contact angles, even with the limited accessibility of many microfluidic structures.

Microelectronic Cooling

As microelectronic devices have been scaled to smaller dimensions and higher speeds, power dissipation has become a major challenge. This problem is now being compounded by the move to extend the conventional planar architecture that has dominated integrated circuit design for decades into the third dimension by stacking completed circuits on top of the other. Although the approach has obvious benefit in increasing the density of computing power, it also multiplies the electrical power that must be removed.

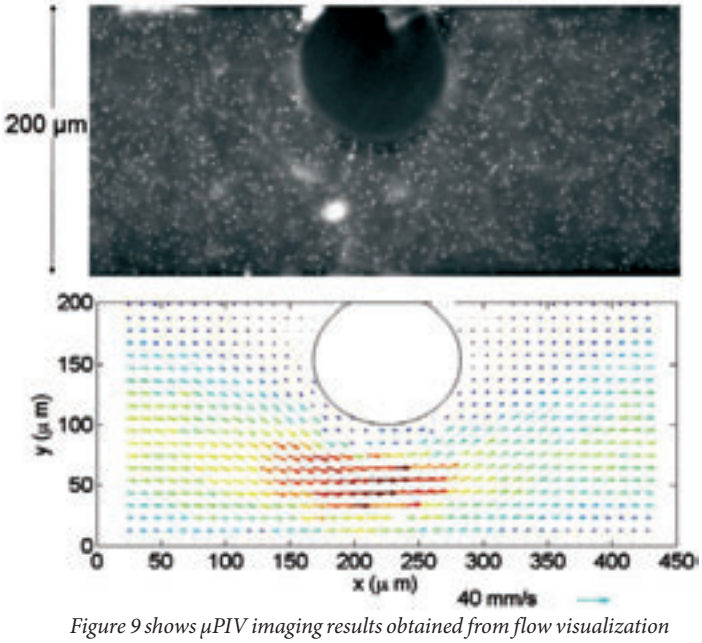

Figure 9 shows µPIV imaging results obtained from flow visualization around a growing bubble. The tracking particles used for micro PIV are clearly visible in the image at the top. The arrows in the lower plot indicate the speed and direction of flow across the two-dimensional section defined by the focal plane of the imaging system. Since the imaging system is not sensitive to movement in the Z-direction, some of the errors in the 2D field are associated with the projection of the true, three-dimensional velocity vectors onto the plane of the image. In theory, ACM could support three-dimensional micro PIV measurements, if images can be acquired with sufficient speed.

Currently, the most common approach to three-dimensional interconnect architecture is simply stacking the chips within a package and connecting them with wire bonds. A number of other techniques are in development that will connect multiple dies with through-silicon-vias (TSV) that pass through the chip itself. Some experts have speculated that we may eventually be able to connect as many as a hundred circuits in three-dimensional integrated circuits (3DIC), providing enormous increases in computing power. Clearly, a successful heat dissipation strategy is a prerequisite.

One solution currently under consideration is the incorporation of microchannels in the interconnect architecture to conduct cooling fluid through the circuit. The cooling efficiency of such an approach can be enhanced if the fluid boils in the process, thereby taking advantage of the latent heat of the phase transition to increase the total removal. However, the formation of bubbles in the boiling fluid also has the potential to interfere with cooling, if the bubbles, which nucleate on the channel walls, grow large enough to block the flow before they detach and become entrained in the flowing liquid.

Ongoing research at the MHTL focuses on the micro scale physics of bubble nucleation, growth and detachment. If researchers can understand the forces exerted on a bubble by the fluid that lead to the bubble's detachment, it may be possible to determine conditions that will prevent the formation of large bubbles and ensure reliable cooling.

Conclusion

Though microfluidic devices are already commonplace in applications ranging from ink jet printers to labs on chips, much work still remains to gain a detailed understanding of fluid behavior on the microscale. New applications that could have far reaching effects include fluid transport in fuel cells and cooling in microelectronic devices. Both of these involve these use of two-phase flows in which three dimensional phenomena play an important role. Advanced confocal microscopy can provide fast, three-dimensional visualization of both structure and flow at the micro scale and promises to make significant contributions to the continuing development of microfluidic technologies. ■

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