

Materials for Micro- and Nanofluidics

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Guest Editors

Abstract

Over the last two decades, our ability to create networks of fluidic channels of sub-millimeter or even sub-micrometer diameters has led to a wide range of microchemical applications. Whereas early efforts were directed toward the development of microanalysis systems, in more recent times the development of microreactors and tools for biotechnology and basic biological studies has emerged. This issue of *MRS Bulletin* highlights the many different ways in which material properties are crucial in the fabrication, assembly, and operation of micro- and nanofluidic systems. Choice-of-material considerations range from an assessment of whether a desired channel design can be microfabricated in a certain material to whether the material is compatible with the operating conditions (i.e., pressure, temperature) and the chemical composition (solvent, solutes) of the fluid used. Moreover, in certain cases, specific surface or bulk material properties can be used to the benefit of the application of the device. In the development of today's wide range of integrated micro- and nanofluidic applications, a common challenge emerges: meeting the often contradictory set of constraints imposed on the physical and chemical properties of materials by the envisioned applications. This issue reviews these challenges and their solutions and provides an outlook on how the ingenious use of existing and new materials can spur the development of ever more sophisticated micro- and nanofluidic systems.

Keywords: biological, fluidics, nanoscale.

Introduction

Micro- and nanofluidic systems have been around as long as the earliest cellular forms of life on our planet. In many biological species, including humans, microvascular systems—networks of convective flows that permeate their volumes—provide mass transfer of chemicals to and from certain locations. Early examples of artificial micro- and nanofluidics include micro syringe needles, glass capillaries, and a wide variety of membranes with micro- or nanopores. Advances in micro- and nanoscale fabrication and patterning methods have opened routes to attain micro- and nanofluidic networks of more sophisticated design and interconnectivity only over the last two decades. Three main categories of applications have inspired these rapid developments:

1. *Microanalysis systems* for purification, separation, and/or identification purposes; this was the original theme of microfluidics launched by Manz, Harrison, and Ramsey in the early 1990s¹ and has

led to a variety of clinical and diagnostic tests, chemical and biological agent detectors, and environmental tests.

2. *Microreactors* for chemical synthesis; this younger field, which started in the late 1990s, has brought us, for example, methods to synthesize dangerous/unstable/precious compounds on demand,^{2,3} fuel processing systems,⁴ synthesis of tailored nanoparticles,⁵ and microfuel cells.⁶

3. A wide range of *enabling microfluidic tools* for biological, biotechnological, and biomedical applications; these tools have burst on the scene over the past decade for purposes ranging from interacting with single cells⁷ to screening for appropriate crystallization conditions for proteins.⁸

Characteristics of the Microscale

Before venturing into the materials aspects of the design, fabrication, and use of different micro- and nanofluidic chips, let's first consider the inherent implica-

tions of performing chemical and biological processes in networks comprising channels of sub-millimeter dimensions. First, *fluid flow is laminar* (i.e., no turbulent mixing), as flows in micro- or nanofluidic channels are dominated by viscous dynamics, due to their higher surface-to-volume ratio, and are characterized by a Reynolds number ($Re = Vh/\nu < 100$, where V is the average velocity of the flow in meters per second, h is the characteristic cross-sectional dimension of the channel in meters, and ν is the kinematic viscosity of the fluid in square meters per second). Due to the lack of turbulence, mixing of fluids is controlled by diffusion only. In laminar flows, the pressure required to push fluids through a channel scales with $1/h^2$ for a fixed velocity; depending on the flow speed and size of the network, this relationship may lead to pressure requirements that are experimentally inaccessible, for example, because of material failure.

Second, the smaller dimensions lead to *higher rates of mass and heat transfer* as a result of the steeper concentration and temperature gradients.

Third, compared with the macroscale, micro- and nanofluidic channels have *very high surface-to-volume ratios*. Having high surface-to-volume ratios enables, for example, the use of electro-osmotic flow—flow driven by the interaction of an electric field with the net charge adjacent to a charged surface—rather than pressure-driven flow. On the other hand, higher surface-to-volume ratios may be detrimental for certain processes because of nonspecific adsorption, most notably in applications involving proteins.

In any micro- or nanofluidic application, some or all of these microscale characteristics have to be accounted for—or, ideally, exploited—in the design and function of devices.

Microfabrication Methods

The wide range of applications for micro- and nanofluidic networks and the characteristics of the microscale impose a wide variety of constraints on the materials used in their fabrication. Historically, the first micro- or nanofluidic systems were fabricated in silicon using the fabrication methods developed in the semiconductor industry for microelectromechanical systems (MEMS).⁹ The combination of photolithography and wet anisotropic etching allows complicated channel geometries to be created. Glass, Pyrex, and quartz have also long been substrates of choice, due to their chemical inertness and optical transparency. Glass offers an example of a fabrication constraint associ-

ated with a material property: the isotropic character of glass etchants constrains the geometries that are accessible for channels formed in glass; high-aspect-ratio structures are difficult to form. On the other hand, researchers have taken advantage of the isotropic etching properties of glass to form channels with complex cross sections, for example, a controlled undercut of the masked areas.¹⁰ Ease of fabrication has led many researchers to pursue the use of polymeric materials, shaped into microfluidic networks using various molding strategies. The elastomer poly(dimethylsiloxane) (PDMS)—popularized by Whitesides,¹¹ Beebe,¹² and many others—is one of the more commonly used polymers in microfluidics. The desired channel designs can be obtained by replica molding from a master defined in standard photolithographic procedures. The resulting cross-linked material is highly transparent and seals well against itself and other surfaces, and the integration of pressure-fit interconnects is straightforward. Incompatibility with most organic solvents, however, has limited the use of PDMS to aqueous-based applications.

In addition to silicon, glass, and PDMS, many other materials, other fabrication methods, and combinations of materials are being used. One up-and-coming microfabrication technique is laser micromachining. With two or three different laser sources (e.g., a combination of a 197-nm Ti:Sapphire laser and a 1064-nm YAG laser), microfluidic channels can be machined in the surface of just about any material with a precision of 1 μm or less.¹³ The key disadvantages of laser micromachining are its high equipment cost and the serial (and thus slow) nature of the process, whereas techniques based on photolithography, etching, or molding are parallel in nature.

Choice of Materials

Researchers often base their choice of materials on their familiarity with them and the associated methods of fabrication, or on the availability of appropriate fabrication facilities. With the field moving away from the fabrication and characterization of individual components toward a focus on the fabrication and application of *integrated* microchemical systems for specific chemical and biological purposes, decisions on what material or combination of materials to use must be based on the often stringent requirements of the envisioned application.

Material properties that have to be taken into account when making these decisions include:

- Compatibility with operating conditions (e.g., pressure, temperature), media, chemistries, and biological entities;
- Surface properties: roughness, zeta potential, wettability, bio-adhesion;
- Bulk properties: mechanical strength, transparency, thermal expansion, permeability; and
- Ease of integration with other materials: bonding/sealing possibilities.

In their article in this issue, Zhang and Haswell include a table that compares a range of these properties for the most frequently used materials for the fabrication of micro- and nanofluidic networks.

A DNA analysis device (Figure 1), as reported by Burns et al. in 1998, was one of the very first examples of a truly integrated microfluidic system.¹⁴ Burns's work exploited the wetting characteristics of his substrates—glass and silicon—to drive and regulate flow by capillarity. Another example of an integrated microchemical system in which material properties played a crucial role is Motorola's micro fuel processor for the conversion of methanol at 250°C into hydrogen for a fuel cell.⁴ A

fuel processing chamber, a catalytic combustion chamber, a fuel vaporizer, resistive heaters, pressure and temperature sensors, and other components are integrated in layers of ceramic tape that are sintered together into a single fuel processing unit. Ceramics are known as excellent substrates to serve as catalytic supports. Key material challenges included matching thermal expansion coefficients and bonding dissimilar materials to deal with heating and cooling cycles.

From our own work (Kenis), we illustrate meeting combinations of material requirements for a microfluidic application through the synthesis of ceramic catalyst support structures for hydrocarbon reforming.¹⁵ To achieve high hydrocarbon-to-hydrogen conversion per volume while avoiding coking of the catalyst (which takes place up to about 750°C), we set ourselves the following requirements: (1) surface area of at least $5 \times 10^5 \text{ m}^2/\text{m}^3$; (2) thermal stability above 800°C; and (3) a high porosity to produce a low, experimentally readily achievable pressure drop across the structure. Whereas the high-

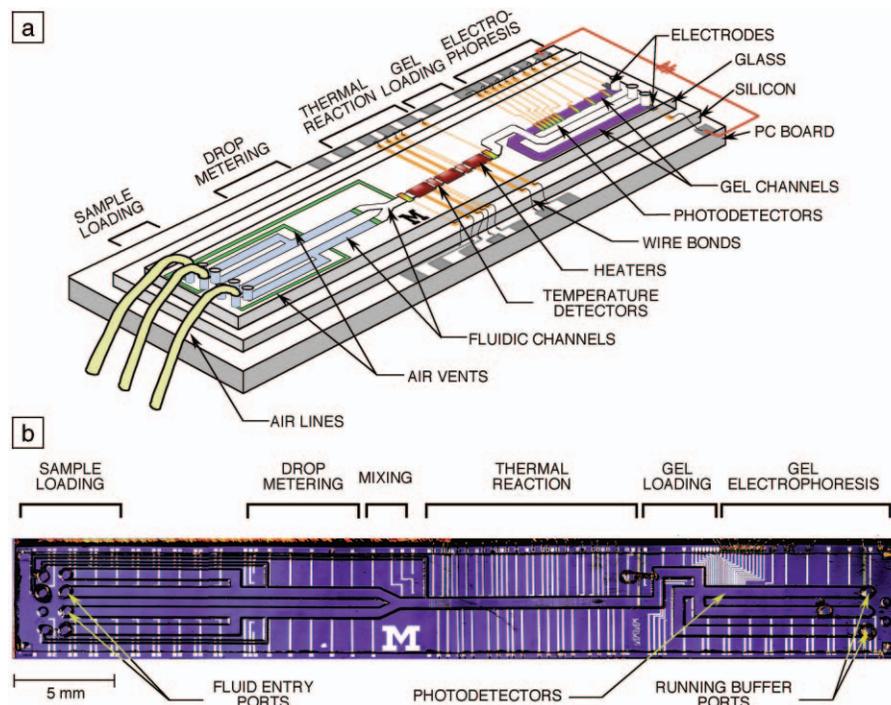


Figure 1. (a) Schematic illustration of an integrated microfluidic device with two liquid samples and electrophoresis gel present. The only electronic component not fabricated on the silicon substrate, except for control and data-processing electronics, is an excitation light source placed above the electrophoresis channel. Color code: blue, liquid sample (ready for metering); green, hydrophobic surfaces; purple, polyacrylamide gel. (b) Optical micrograph of the device from above. Wire bonds to the printed circuit board can be seen along the top edge of the device. The color arises from the interference filter reflecting the short-wavelength light. The pressure manifold and buffer wells that fit over the entry holes at each end of the device are not shown.¹⁴

surface-area features of oxides such as anodized alumina typically do not survive temperatures above 700°C, the non-oxides, such as zeolites, typically do not meet the low-pressure-drop requirement. Figure 2 shows the highly porous, inverted beaded structure made out of SiC that we synthesized by infiltrating beds of packed polystyrene spheres with a pre-ceramic polymer, followed by curing and sintering.¹⁵ Such SiC or SiCN catalyst support structures have surface areas as high as $7.4 \times 10^7 \text{ m}^2/\text{m}^3$, are stable up to at least 1200°C, and have a porosity of ~ 0.7 , which ensures a low pressure drop. The integration of these catalyst structures into alumina reactor housings leads to integrated microfluidic reactors for hydrogen generation at $>99.8\%$ conversion using ammonia as the fuel.

Many microfluidic designs are capable of performing only a few tasks at the same time. Performing many tasks in parallel, as desired in screening tools, was made possible by the advent of very large-scale integrated microfluidic networks (VLSI-mFNs) of PDMS (Figure 3) in which the elastomeric properties of PDMS are used to create arrays of integrated pneumatic valves that can also be used to create pumps and multiplexers all fabricated out of the same material.^{16,17} In each valve, two channels cross but are separated by a thin membrane of PDMS. If one of the channels is pressurized, the membrane bulges out into the second channel and closes it off (like stepping on a garden hose). Consecutive actuation of three pneumatic valves creates a peristaltic pumping mechanism. Such VLSI-mFNs have been used, for example, for DNA sequencing¹⁸ and protein crystallization.¹⁹ A startup company, Fluidigm, has already introduced VLSI-mFN-based microfluidic systems to the market that screen for suitable crystallization conditions.²⁰

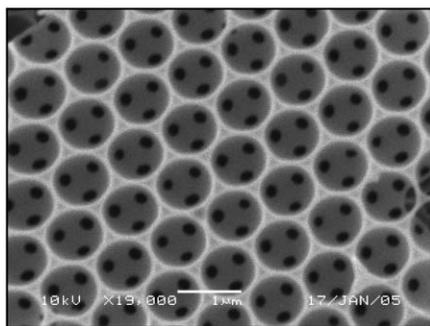


Figure 2. Scanning electron micrograph of a highly porous SiC catalyst support structure with thermal stability to over 800°C.¹⁵

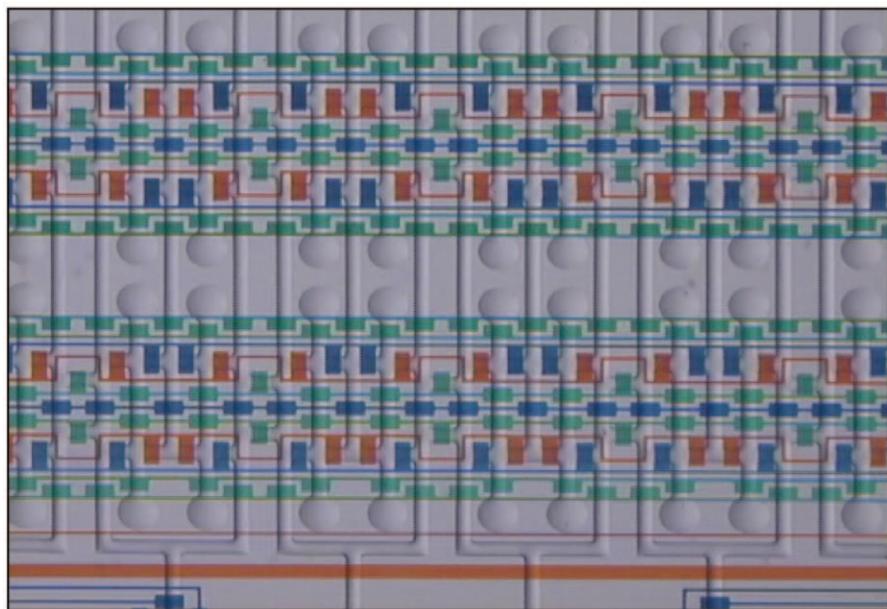


Figure 3. Optical micrograph of part of a very large-scale integrated microfluidic network (from S. Quake's research group, Stanford University): Three independently addressable networks (red, blue, green) of pneumatic valves (rectangles connected via very thin channels) enable pumping of fluid through the microfluidic channels (filled with clear solution) by consecutive actuation.^{16,17} The fluid channels are 50–100 μm wide. Photo by Sebastian Maerkl.

Micro- and nanofluidic systems have also been extensively integrated with electronics and photonics for detection and actuation purposes. In a recent report, a microfluidic device with integrated chemical sensing and electrowetting actuation on chemoreceptive neuron metal oxide semiconductor transistors was reported.²¹ Chemical sensing can also be accomplished through the integration of photonic crystal-based nanolasers.²²

Several additional examples of integrated micro- and nanofluidic systems will be covered in the articles that follow this general introduction. In picking topics for these articles, we focused on covering a wide range of different materials, including silicon, glass, polymers, and hydrogels, as they are currently being used in micro- and nanofluidics. As the reader will see, the wide range of potential applications for microfluidic systems, ranging from analysis systems for pathogen detection to chemical synthesis and spectroscopic study tools, has been made possible by these developments. Each article discusses micro- and/or nanofluidic applications in light of various materials aspects, including the various compatibilities (conditions, chemistries), integration/bonding challenges, surface properties, and bulk properties briefly mentioned previously.

Zhang and Haswell provide a general review of how materials matter in the fabrication and operation of microfluidic systems. They look in more detail at the most popular materials and methods currently being used for chip fabrication. They also provide a treatment of how the high surface-area-to-volume ratios of microfluidic systems have been and can be exploited for various purposes.

After introducing the various advantages and disadvantages of metals, ceramics, glass, and polymers for various microfluidic applications, Jensen focuses on the use of silicon as the material of choice for a variety of applications: these range from chemical synthesis (screening of reaction conditions, synthesis of quantum dots) to heterogeneous catalysis (hydrocarbon fuel conversion, hydrogen purification). The challenges involved in the fabrication and operation of hybrid structures composed of more than one material are exemplified by the integration of various sensors (pressure, temperature, chemical composition) and meeting the requirements of optical transparency for spectroscopic applications (UV, IR).

Bakajin and Austin compare the benefits and drawbacks of elastomeric and non-elastomeric materials in the development of micro- and nanofluidic devices for DNA/RNA analysis and protein-folding

studies. The authors provide a primer on the considerations of surface forces and solid mechanics in choosing materials for micro- and nanofluidic systems. Additionally, they emphasize the specific challenges that arise when precise submicrometer structures are required, and when applications using living cells are involved.

Stroock and Cabodi review initial efforts to implement microfluidic networks as synthetic vascular systems to control the chemical state inside materials used in biomedical applications. They highlight challenges that arise in microfabrication involving soft, organic biomaterials. Using tissue engineering and wound treatment as target applications, they illustrate basic design considerations that are relevant to the creation of these chemically active materials. Finally, they point toward the outstanding challenges in this emerging application of microfluidic technology.

Lastly, Simmons et al. discuss the fabrication and performance of microfluidic analysis systems in which species are separated using insulator-based dielectrophoresis (iDEP). These chips may find application in pathogen/bacterial analysis, sample preparation in microanalysis systems, and biomedical diagnostics. Their theoretical and experimental comparison of surface and bulk material properties shows how the choice of material (glass or polymer) can have a significant effect on throughput, detection efficiency, and cost of the microfluidic application.

Current and Future Challenges

The field of microchemical systems is increasingly focused on the creation of integrated systems for specific chemical or biological applications. Their development requires a balancing act to meet the often contradicting material requirements for the specific series of tasks. The lack of materials with specific combinations of properties also encourages innovation. For example, multiple new surface-modification approaches and bonding/sealing methods have emerged. Also, the incompatibility of PDMS with many organic solvents (which can cause swelling and dissolution) has spurred the development of perfluorocarbon-based elastomeric materials²³ that are more compatible with typical organic solvents. Another emerging direction is the development of methods to fabricate arbitrary three-dimensional

networks. Until recently, most approaches to obtain “3D” microfluidic networks involved sealing of layers, resulting in “2.5D” networks—essentially a series of connected 2D layers. Recently, the fabrication of 3D networks with arbitrary microfluidic interconnectivities using a direct-write approach has been reported.²⁴ In addition, Ke et al. have recently reported another promising method to directly machine nanofluidic channels inside glass substrates through the use of a highly focused laser beam.²⁵

With researchers in academia and industry pursuing such a wide range of applications for microchemical systems, the rate of novel, ingenious materials solutions being reported will only increase. This issue of *MRS Bulletin* will review the wide range of materials aspects that need to be taken into account in the development of micro- and nanofluidic applications. Many examples are described, including several in which novel material solutions were crucial to their inception. We hope that these articles provide not just a good overview of the central role of materials in the development of microchemical systems, but also spur ideas for the ingenious use of new materials and methods of processing to allow for ever more integrated micro- and nanofluidic systems.

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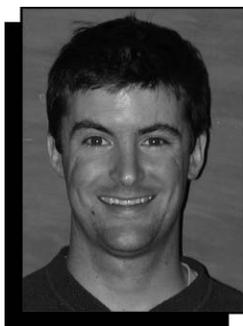
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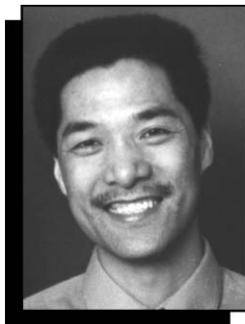
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