Purifying nanomaterials with a nanomaterial

TECHNOLOGY ADVANCES seeks materials developments on the threshold of commercialization.
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The pitch

Commercial separation membranes with pores in the size regime of ~5–50 nm are typically polymeric materials that are micrometers thick, which leads to pore morphologies that resemble long narrow tubes or tortuous-path three-dimensional matrices. SiMPore, Inc. offers a new class of porous membranes (UltraSM®) that combines nanoscale thickness of <20 nm with macroscopic yet robust millimeter-scale lateral dimensions and tunable pore sizes (see Figure 1). As the membrane thickness approaches the pore diameters, a simplified structure of holes in a thin sheet results, greatly enhancing both diffusive and forced flow through the membrane. The precisely controlled maximum pore size enables a sharp size cutoff between molecules that will pass through the membrane and those that are blocked.

The molecular separation markets are currently served by conventional polymer membranes at the low end and electrophoresis and chromatography at the high end. Each has significant limitations that SiMPore's ultrathin nanoporous silicon membranes solve efficiently and economically. Compared to conventional membranes, SiMPore's membranes are ~1000 times thinner, resulting in less sample loss and higher flow rates. In addition, the sharp pore size cutoffs provide greater separation precision. Compared to chromatography, SiMPore's membranes are less expensive, usually faster and easier to use, and require little post-separation processing. In effect, these membranes fit in the "sweet spot" between conventional membranes and chromatography, as shown graphically in Figure 2.

The technology

The UltraSM membrane material, porous nanocrystalline silicon, is now being scaled up to commercial volumes (see Figure 1b). The porous membrane is formed on a silicon wafer substrate when a thin layer of sputter-deposited amorphous Si is crystallized in a highly controlled rapid thermal process. Pores are formed in <1 min as nanocrystals nucleate and grow in a thin amorphous Si film as complex strain fields are created. Thus no costly nanolithographic patterning technologies are required to define the pores. Pore size and density are indirectly controlled through deposition and annealing parameters. Appropriate masking and etching processes allow comparatively large membrane areas to be freely suspended across openings in the silicon wafer frame with geometries that meet the requirements of each targeted application. Scaling up the production of this nanomaterial is simplified by the vast existing silicon wafer processing infrastructure (i.e., deposition, thermal, etch, patterning, and cleaning).

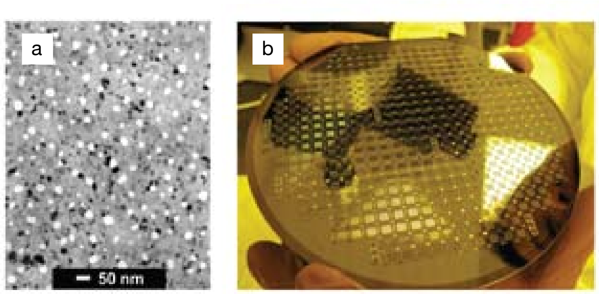

Figure 1. A transmission electron micrograph of (a) an ultrathin porous nanocrystalline-Si membrane. In this bright field image, the pores appear as white spots, while the nanocrystalline silicon matrix is grey with black dots corresponding to the fraction of nanocrystals in a diffracting orientation. (b) A 150 mm wafer with a several-hundred membrane die, ready for integration into SepCon centrifuge devices.

Beyond separations, SiMPore's membrane technologies can be applied in a variety of markets in electron microscopy where thin electron-transparent materials are critical substrates. In cell culture applications, an artificial barrier like the UltraSM membrane that is approximately the same thickness as a cell membrane offers substantial benefits for monolayer drug permeability studies to assess drug effectiveness, cell co-culture studies of interaction and communication between cell lines, and *in vitro* tissue assays. Commercial cell growth membranes are micrometers thick, creating a significant barrier to molecular transport in these applications and also making fluorescent and high-resolution optical imaging of cells very difficult. The UltraSM membrane is nearly transparent optically, thereby enabling high-quality imaging of cells on both sides of the membrane with no fluorescence background.

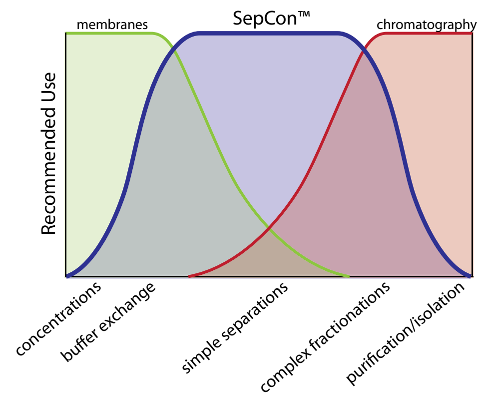

Figure 2. The characteristics of SiMPore UltraSM membranes enable meeting a variety of molecular separation needs that are not adequately served by existing membrane technology or more complex chromatography processes.

Opportunities

SepCon™, SiMPore's first separations product line, will debut as a membrane insert for 1.5 mL centrifuge tubes to be released through the company's e-commerce Web site in December 2010. SiMPore is seeking strategic partners and potential licensees to assist the company with follow-on product development and market penetration. The company's core strength and focus is its novel membrane material and fabrication capability; therefore, partners with expertise in integrating membranes into various products for life science research, drug development, and healthcare markets would create particularly attractive opportunities.

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High-temperature polymer for electronics and medical device applications

The pitch

Challenges are mounting for increasing the reliability of electronics and medical devices and protecting them from the detrimental effects of both physical and operational environments. Among the several desired areas of improvements, manufacturers of electronics and medical devices are seeking protective materials, preferably in the form of thin film coatings with improved processing characteristics, higher temperature integrity, excellent barrier properties, corrosion resistance, suitability at high-frequency operations, and even better dielectric properties.

Parylene (p-xylylene) polymers have been valued by the electronics and medical devices industries for their inertness, gas phase deposition, absence of pinholes, and excellent barrier properties. Parylene coatings are being used in numerous implantable devices including pacemakers, implantable cardiac defibrillators, coronary stents, cochlear and ocular implants, and neurostimulators. External devices such as transdermal drug delivery devices, digital dental imaging systems, and surgical and endoscopic devices, for example, have also benefited from Parylene's lubricity and barrier properties. Recently a variety of devices have been created that utilize Parylenes in microelectromechanical systems technology. They have also been incorporated as structural materials for microfluidic devices and are being used successfully in electronics to provide enhanced reliability and a trouble-free life. Because Parylene coatings are applied in very thin layers, heat tends to dissipate rapidly from the underlying electrical components. Thus the coated electrical components cool down quickly and are less prone to temperature-related degradation than similar components bearing other types of coatings.

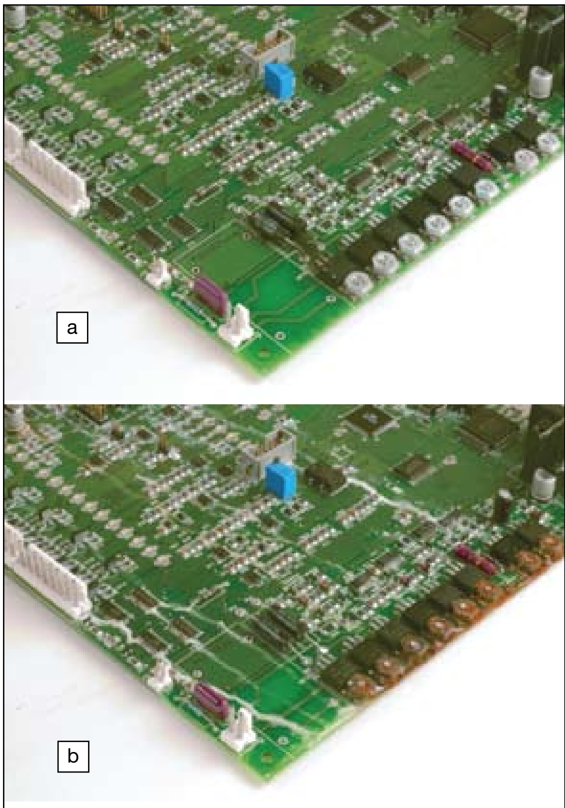
The technology

Parylenes are applied to substrates in a vacuum chamber at pressures of 10^{-5} Torr or below. Parylene coatings are formed at approximately 0.1 Torr. Under these conditions, the mean free path of the gas molecules in the deposition chamber is on the order of 0.1 cm. As a result, all sides of an object are uniformly impinged by the gaseous monomer providing a high degree of conformability.

Vacuum deposition polymerization begins with the vaporization of a Parylene dimer. The dimer vapor is pyrolytically cleaved to form a reactive monomer vapor. The reactive monomer vapor is then transferred to a deposition chamber where the substrates are located. In the deposition chamber, the reactive monomer vapor spontaneously condenses onto the substrates to form the Parylene coating. The entire process is carried out in a closed system under constant negative pressure. There is no liquid phase in the deposition process and the substrates remain near ambient temperatures.

Parylene HT, a fluorinated variant of Parylene by Specialty Coating Systems, is engineered to provide long-term thermal stability up to 350°C (662°F), with intermittent exposures up to 450°C (842°F) and provides a pinhole-free barrier to protect against various fluids as well as moisture, chemicals, solvents, acid, and common gases; resists chemical attack; and is insoluble in all organic solvents, mineral acids, and bases at room temperature.

Parylene HT successfully resists the corrosion of many electronic assemblies, medical devices, and substrates in a variety of application conditions. Since the actual corrosive environment changes from day to day (or faster), most testing experiments are designed to expose samples to various environmental stresses that promote corrosion. The salt fog test is an accelerated corrosion test in which specimens are exposed to a fine mist of a solution that usually contains sodium chloride (typically 5%). Several printed circuit boards coated with Parylene HT along with controls were subjected to 144 h of salt fog exposure. Parylene HT-coated boards did not show any evidence of corrosion while the uncoated boards had salt and corrosion residue running vertically down the board as shown in the figure.



Close up view of (a) a Parylene HT-coated and (b) a corroded uncoated printed circuit board after salt fog test.

Additional advantages of Parylene HT coatings include high resistance to oxidative degradation when exposed to ultraviolet radiation; the lowest dielectric constant and dissipation factor among the Parylenes enabling it to provide small, tight packages with dielectric insulation through a thin coating; and excellent dry-film lubricity (coefficient of friction is 0.15, which is about half that of many polymers including other Parylenes).

Opportunities

Specialty Coating Systems is seeking to develop advanced applications for Parylene HT coatings for medical and high-temperature electronics.

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Closed-cycle optical cryostat as an integrated system

The pitch

Optical cryostats are used to lower the temperatures of experimental samples for materials research. This can be accomplished by using the cryogen, liquid helium, or using closed-cycle techniques. However, liquid helium is becoming costly and difficult to purchase, which encourages the use of closed-cycle refrigerators. In addition to not requiring the cryogen, closed-cycle systems can be simpler and easier to use and maintain. Cryogen-free optical cryocooled products currently offered either isolate the sample from vibrations at the expense of operating at higher temperatures or they achieve low temperatures (4 K) but do not effectively isolate the sample from vibrations. Potential users have to select one or the other or return to using an open-cycle helium-consuming solution. In addition, some of the best current solutions require the researcher to create elaborate installations in order to isolate the sample from vibrations.

New technology developed by Mountain Instruments allows low temperature and low vibration as well as greatly simplifying the operation of the cryogenic system (see figure).

This sample space stability enables research that is not currently possible in closed-cycle cryocoolers, thereby opening new market opportunities. The market will also benefit from the integrated solution that can save researchers laboratory time by simplifying setup and eliminating fine-tuning. Currently the market size for optical cryostats exceeds $50 million. This new technology should enable more applications and capture the market share of those university researchers and government laboratories seeking reduced helium costs and increased operational efficiency.

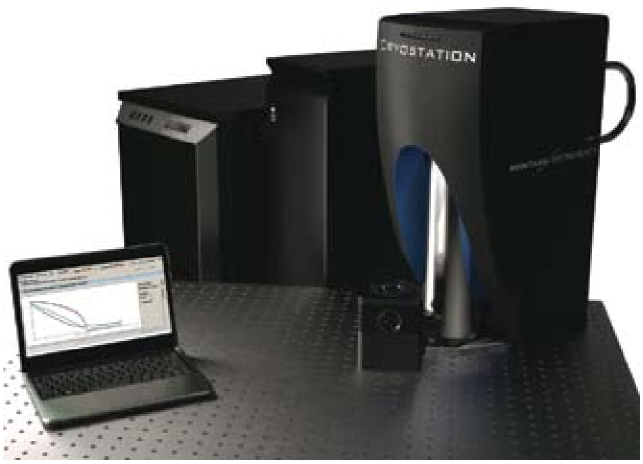
The technology

With this new technology, vibrations are managed using a combination of proprietary thermal conductors, a symmetric opposed bellows design, and spring dampening components. The linkage from the cold finger to the sample platform uses a propriety material that was optimized to transfer heat efficiently at low temperatures and also allows large temperature gradients across it. With this, the sample temperature can be increased to 350 K while the cryocooler stays cold. This vibration-isolated architecture makes it possible to mount the sample firmly to the table like other optical elements, yet have the cold source literally float on a spring mechanism nearby. A balanced bellows design allows the floating section to transfer very little force to the sample area.

The result is that the Cryostation reaches temperatures of 3 K while isolating the cooled sample from vibrations sufficiently well to sustain typically <2 nm of vibration. In addition, the Cryostation isolates the cryocooler motor from both the sample and the optics bench it is mounted on so that compressor, motor, and pressure pulse vibrations do not affect the surrounding optics and equipment.

Montana Instruments has also created a new component for thermal dampening called HeatCap, which is a matrix of high-conductivity and high-heat capacity materials. The resultant material acts like a thermal capacitor with the capability to store and release heat quickly with virtually no thermal resistance. As the cryocooler temperature varies by hundreds of milliKelvin, the thermal capacitor reduces those variations to the sample area. By using temperature sensors on the sample platform and actively managing the heat flow using control loop electronics (patent pending), the sample temperature fluctuations can be less than 1 mK.

The Cryostation completely automates tasks such as pumping down the chamber to the appropriate vacuum, checking for leaks, purging with an inert gas after warm-up, thermometry calibrations, and many critical self-check functions. Large temperature-range data sets, which are typically time-consuming, can be acquired automatically. Functions are controlled and monitored through a simple graphical user interface on a mini-notebook computer. The existing sample space in the Cryostation allows performing both spectroscopy applications by using the radial access ports and microscopy applications by using the top and bottom access ports. High pressure, magnetic field, large samples, and many other applications are possible by utilizing the large sample space. Montana Instruments is planning to introduce more application-specific sample areas in the future.



The whole Cryostation system.

Opportunities

The Cryostation is a completely integrated tool that is available for researchers now. The patent-pending technology for managing heat and vibration are available for licensing and or collaborative research. The cold platform is available for integration into high-performance imaging products and solutions.

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