New imaging tool directly measures liquid surfaces

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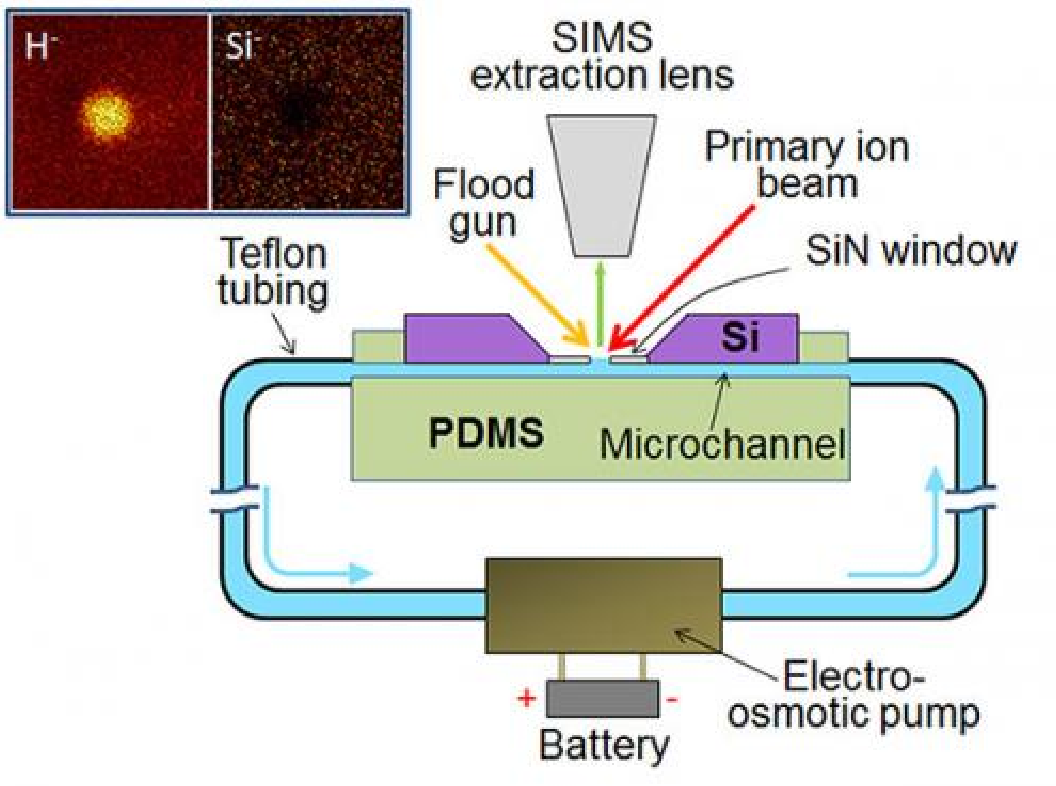

The schematic of a vacuum compatible aqueous SIMS device. Insert shows the secondary H- and Si- ion images of this aperture.

(Phys.org) —A unique chemical imaging tool readily and reliably presents volatile liquids to scientific instruments, according to a team including Pacific Northwest National Laboratory. These instruments require samples be held in a vacuum, which is often incompatible with hydrocarbons and other liquids. Designed and built at PNNL, this one-of-a-kind sample holder continuously pumps the liquid through a gold-coated microfluidic chamber. The extremely narrow channel provides high linear velocity at the detection window and helps overcome the liquids' tendency to vaporize. Instruments access the liquid via an open viewing port. Tests with electron microscopes and mass spectrometers prove the device can operate continuously for up to 8 hours. Further, the device handles complex liquids.

"Great discoveries often require great tools," said Dr. Louis Terminello, who leads chemical imaging work at PNNL. "The discoveries needed to solve today's problems aren't something that you're going to get by eyeballing a sample and analyzing a spreadsheet."

Quick, affordable methods of storing electricity from wind turbines. Effectively trapping and removing industrial solvents from soil. These and other endeavors need precise knowledge of interfacial reactions, the interactions that occur between a liquid and solid or two liquids. This study demonstrated that the PNNL-created device allows surface tools such as microscopes and secondary ion mass spectrometers to obtain the needed data on samples and providing answers scientists need.

"Our device opens a window to observe interactions between liquid/solids and liquid surfaces, which are relevant to liquid/solid heterogeneous catalysis and energy storage techniques," said Dr. Xiao-Ying Yu at PNNL, who worked on the study.

Scanning electron microscopy, time-of-flight secondary ion mass spectrometry, and other surface characterization techniques often require the sample to be in a vacuum. Starting in 2010, the team used soft lithography to fabricate the device's microchannel. The channel was coated in gold to reduce the liquid's permeation into the device. A pump forces liquid through the channel continuously. Above the channel is a window open to the instrument's vacuum, allowing the instrument's electron or other beams access to the sample.

"Using our microfluidic device is advantageous compared to certain other techniques as it allows us to observe these complicated systems in their native environments," said Dr. Theva Thevuthasan, who led the research and is part of PNNL's Chemical Imaging Initiative.

The team examined the surface of liquids of increasing complexity. Many of these liquids were provided by SPI Supplies/Structure Probe, Inc. These liquids included organic solvents in aqueous solutions and IgG-conjugated gold nanoparticles in an aqueous solvent. The team used the device with energy-dispersive X-ray techniques in EMSL's scanning electron microscope and in the time-of-flight secondary ion mass spectrometer. The experiments proved the device could provide better detection of nanoparticles suspended in the liquid and identifications of characteristic fragments of IgG in the flow cell compared with conventional dry samples or wet samples in the ESEM mode. Moreover, it can be safely run continuously for 8 hours and could handle high fluence beams for up to 30 minutes.

The microfluidic device is available for use on instruments at EMSL. Researchers from around the world can use EMSL instrumentation free of charge via the DOE user facility's proposal system.

The researchers are reaching out to scientists at universities to get more scientists using the microfluidic sample chamber, which can be applied already in some systems without more development.

More information: Yang, L. et al. 2013. In Situ SEM and ToF-SIMS Analysis of IgG Conjugated Gold Nanoparticles at Aqueous Surfaces, *Surface and Interface Analysis*. DOI: 10.1002/sia.5252

Yang, L. et al. 2013. Performance of a Microfluidic Flow Cell for In Situ ToF-SIMS Analysis of Selected Organic Molecules at Aqueous Surfaces, *Analytical Methods*. 5, 2515-2522. DOI: 10.1039/C3AY26513G

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