The Nanoaquarium –
A Device for *in situ* Electron Microscopy of Processes in Liquids

CNF Project Number: 1542-07
Principal Investigator: Haim H. Bau
User: Joseph M. Grogan

Affiliation: Mechanical Engineering and Applied Mechanics, the University of Pennsylvania, Philadelphia, PA, USA
Primary Source of Research Funding: National Science Foundation-CBET 1066573 and NSF CMMI 1129722
Contact: bau@seas.upenn.edu, joegrogan@gmail.com
Website: http://bau.seas.upenn.edu
Primary CNF Tools Used: Chemical-mechanical polisher, direct wafer bonder

Abstract:

The nanoaquarium is a nanofluidic platform for *in situ* electron microscopy of objects and processes in liquid media. The nanoaquarium consists of a hermetically sealed, thin (~100 nm tall) liquid cell sandwiched between two electron-transparent silicon nitride membranes. The nanoaquarium is equipped with micro patterned electrodes. The device has been used to image electrochemical deposition and etching, growth and dissolution of nanoparticles, diffusion limited aggregation, crystallization, nucleation and bubble growth, nanobubble migration, interfacial phenomena, to assess the interactions between electron radiation and fluids, and to pattern nanostructures without a need for a mask.

Figure 1: A photograph of the nanoaquarium (top) and a schematic of the nanoaquarium's cross-section (bottom).

Summary of Research:

The nanoaquarium is a custom-designed, microfabricated, nanofluidic device for *in situ* electron microscopy of processes taking place in liquids and of objects submerged in liquids (Figure 1). Two thin (<50nm), electron-transparent silicon nitride membranes sandwich a thin liquid layer, ranging in thickness from tens of nanometers to a few microns. The cell is hermetically sealed from the vacuum environment of the electron microscope. Due to its small thickness, the liquid layer does not significantly scatter electrons, allowing nanoscale resolution imaging of objects suspended in the confined liquid. The nanoaquarium contains electrodes patterned on the silicon nitride membrane for electrochemical measurements and for actuation.

The fabrication of the device was accomplished with direct bonding of silicon wafers coated with silicon nitride. One of the wafers contains a thin film of patterned silicon oxide that defines the shape and height of the imaging chamber and the liquid conduits. The use of direct wafer bonding eliminates risk from potential contamination from glue, epoxy, and other sealing materials and assures a hermetic seal. Use of a dielectric material as the spacer allows electrodes to be directly incorporated into the device. A schematic of the nanoaquarium is depicted in Figure 1. The fabrication process has been previously described [1]. Our lab, in collaboration with others, has used nanoaquariums to study nanoparticle aggregation and colloidal crystal growth dynamics [2-4]; interactions of nanoparticles with moving interfaces [5]; electron beam-induced radiolysis [12-14]; bubble nucleation, growth and detachment [6,10,12]; crystallization [8,16]; and electroplating [9,11,17]. We have also demonstrated the use of the electron beam as a "pen" to pattern nanowires without a need for a mask [12]. The device was patented [18].
Since in situ electron microscopy produces large quantities of data, we have developed automated image processing algorithms to extract useful quantitative measures of the observed phenomena [17,19].

In our experiments, we observed spontaneous motion of bubbles. Interestingly, the electron beam plays a dual role in these experiments. E-beam induced radiolysis generates gas that induces bubble nucleation and growth and the beam provides a means to image the bubbles. To better understand and interpret our experimental data, we constructed a model, based on Blake-Haynes theory and predicted bubbles’ shapes, growth rates, and migration. Consistent with experimental data, our model predicts that in the presence of confinement, growth rates are orders of magnitude slower compared with bubbles [20]. Figure 2 compares the predicted and experimentally observed velocity of bubble’s center of mass as a function of time.

Acknowledgements:
Electron microscopy was performed at the Penn Regional Nanotechnology Facility at the University of Pennsylvania and at the IBM T.J. Watson Research Center.

References: