

# Fabrication and Characterization of a Nanonozzle

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## Abstract:

A new fabrication process proposed by the Nanotechnology and Interdisciplinary Research Team (NIRT) uses a nanonozzle as the mechanical interface with liquid applied at high pressure through a mechanical pump. This process has the potential application in direct substrate surface modification. This paper introduces the prototype nozzle design and experimentation of the nanonozzle.

The initial orifice of the nanonozzle was designed with a feature size in a few microns range. The orifice feature size was reduced by growing an oxide layer covering the inside of the silicon nanonozzle. Cross-sections of the nanonozzle at various distances from the original orifice were examined with a scanning electron microscope (SEM). The data from the SEM were analyzed to characterize the oxide growth layer inside the nozzle.

## Introduction:

The new era of nanotechnology demands both mechanical and electronic devices to be made with parts in the nanometer scale. NIRT has proposed a new fabrication process in which a nanonozzle modifies or prints nano-device parts directly onto a surface. One of the many challenges lies in the proper design of an applicable nanonozzle.

Several nozzle fabrication approaches have been considered, including conformal electrodeposition of silicon microneedles, micromachined reconfigurable shadow masks, and photolithography patterning with silicon etching and oxidation. The third approach seems most viable in the context of controllable shape and appropriate characteristics to withstand high pressure. Hence, it is the chosen process for the NIRT project.

The approach of photolithography patterning creates nozzles with orifice feature sizes, or diameters, in the micron range. Hence, oxidation of the silicon inside the nozzle orifice is necessary to reduce the feature size to nanometer scale. There are two types

of oxidation processes: wet oxidation and dry oxidation. Dry oxidation uses oxygen gas to oxidize silicon as depicted by the following chemical reaction equation:



Wet oxidation uses steam to oxidize silicon as shown in the following equation:



The major advantage of wet oxidation over dry oxidation is the faster oxide growth rate. Wet oxidation grows  $\text{SiO}_2$  up to 5 times faster than dry oxidation. For instance, after 10 hours of oxidation on the flat surface of a silicon wafer, wet oxidation process grows 2.2  $\mu\text{m}$  of  $\text{SiO}_2$ , while dry oxidation process grows only 0.4  $\mu\text{m}$  of  $\text{SiO}_2$ .

These oxidation rates reflect oxidation on a flat plane. However, the process NIRT proposes involves oxidation inside an open-ended, tunnel-shaped nozzle. Consistency of the oxidation growth rate for these two different geometries may diverge. Hence, it is also required to determine the oxidation rate inside the nozzle's orifice neck.

## Procedure:

The fabrication process of the nanonozzles involved photolithography. After proper mask design and successful translation of the top view nozzle patterns (Figure 1) onto a silicon wafer, wet chemical etching of silicon (KOH) was used to etch the depth dimension of the nozzles. Due to the crystal orientation of the silicon wafer, hot concentrated KOH etched the (100) crystal plane several hundred times faster than the (111) plane. Hence, the initial nozzle's edges were slanted. A second plain wafer was fusion bonded with the first wafer in order to seal the top opening of the nozzles.

The fabricated nozzle structures' orifices with feature sizes ranging from 3  $\mu\text{m}$  to 20  $\mu\text{m}$  were too large for direct nanojet applications. As a result, wet oxidation was used to grow an oxide layer, ranging

from 1-3  $\mu\text{m}$  of thickness, inside the orifice. Three different nozzle batches were oxidized for 5, 12, and 15 hours independently in a Lindberg furnace at 1100°C.

After completing the fabrication process of the nozzles, the Hitachi scanning electron microscope (SEM) was used to examine and measure the orifice dimensions and the oxide thickness. To better characterize the tunnel oxidation, cross-sections of the orifice neck were needed every other few micrometers apart. A polishing machine with slurry sizes of 1  $\mu\text{m}$ , 0.3  $\mu\text{m}$ , and 0.05  $\mu\text{m}$  was used to polish the nozzles.

### Results and Conclusions:

Wide ranges of oxide growth thickness were obtained for all three oxidation cases (5, 12, and 15 hours). Both of the 5-hour and 12-hour experiments produced oxide thickness ranges (0.7  $\mu\text{m}$  to 1.5  $\mu\text{m}$  and 1.5  $\mu\text{m}$  to 2.2  $\mu\text{m}$ , respectively) less than the theoretical flat surface oxide thickness (2.4  $\mu\text{m}$ ). Only the 15-hour experiment produced an oxide thickness range (2.0  $\mu\text{m}$  to 2.8  $\mu\text{m}$ ) that included the respective theoretical oxide thickness (2.7  $\mu\text{m}$ ). However, these ranges of growth thickness measurements were insufficient to support any conclusive analysis. Accurate examinations and measurements of the nozzle orifice's dimensions were limited by the capacity of the SEM (i.e. degree of visibility at high magnification) and the ability to visually distinguish the boundary of oxide layer and silicon layer. Part of the explanation for the deviated oxide thicknesses measurements could be attributed to inconsistent performance (i.e. low visibility at high magnification) and settings (i.e. the orientation of the specimen in the vacuum chamber) of the SEM.

As a result, more future work with controlled experiments and refined measurement tools (i.e. SEM) would be needed to further explore this nozzle design challenge.

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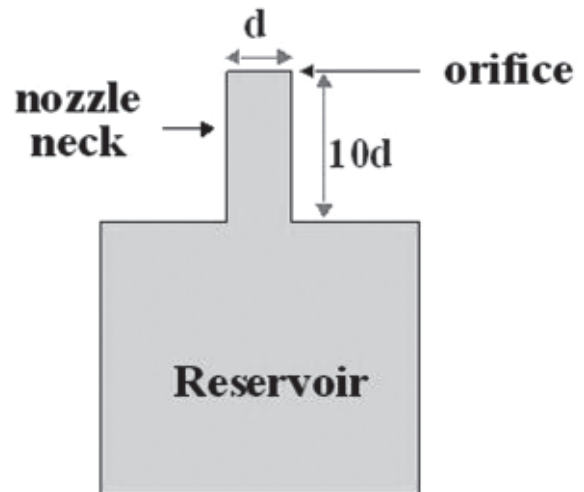


Figure 1: View of nozzle.