Surface Characterization of Etched Micro- and Nano-Structures in Silicon for Phonon Heat Transport

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Introduction:

In insulators, heat is carried by excited vibrational states (phonons) with wavelengths from a few nanometers to several hundred nanometers (nm). Phonons travel through solids ballistically until they are scattered by phonon-phonon interactions, imperfections in the crystal, or boundaries [1]. At low temperatures and device dimensions comparable to phonon wavelengths, surface reflection becomes the primary factor in scattering. Phonons scatter from surfaces with roughness larger than their wavelengths [2]. Because the efficiency of thermoelectric devices is inversely proportional to thermal conductivity [3], surface roughness of devices must be quantified for a better understanding of heat flow.

While the scanning electron microscope (SEM) can be used to visualize topographical outlines and the atomic force microscope (AFM) can be used to measure roughness of near-horizontal surfaces, current microscopy tools lack the capability to measure roughness of vertical or near-vertical sidewalls. The focus of this project was to develop and test a method to characterize the vertical surfaces of nanowalls. Three different techniques were investigated. The most effective method was used to characterize sidewalls created by varying etch processes.

Etched silicon substrates were used.

Experimental Procedure:

Method 1: Break and Tip Walls. Zyvex S-100 tungsten nanoprobes with a tip radius greater than 2 μm installed on the Zeiss Ultra-55 SEM and operating under vacuum during imaging were used. Each probe was placed with 5 nm precision adjacent to a studied silicon nanowall roughly 200 nm thick and 500 nm tall. Piezoelements pushed the probe against the wall with the goal of cracking it at the base and laying it flat for consequent AFM spatial profiling.

Method 2: Tilt Sample in AFM. An aluminum wedge was fabricated to mount samples at 30° to the horizontal in the AFM. The 90° sidewalls therefore became tilted approximately 60° from the horizontal, requiring a tip with an inner half-cone angle less than 30° for successful visualization. NanoScience Aspire CT300 conical tips were used for this purpose. Samples used included 1.8 × 2.2 μm squares etched 200 nm deep into silicon using the focused ion beam (FIB) and silicon mesas etched using the Unaxis 770 deep Si etcher for 5 and 10 minutes generating 1.2 μm and 2.4 μm deep structures respectively.

Method 3: Break and Tilt Substrate. A diamond cutter was used to cleave samples along crystal planes with a precision of approximately 2 mm. Walls sufficiently close to edges of the samples were found using an optical microscope, and samples were mounted vertically using an aluminum block and observed in the AFM using non-contact tips. Samples used had silicon structures etched in a continuous passivation process using the Unaxis 770 deep Si etcher for 5 and 10 min. Walls parallel and at 45° to the cleave planes were imaged.

Results:

Method 1: Break and Tip Walls. The probes were successfully used for cracking pieces of the nanowalls. However, only small sections of walls that had come into direct contact with the probes were knocked over, possibly affecting the observable roughness of the exposed surfaces. Moreover, the pieces were not stationary on the substrate and this method was found to be unsuitable for profiling the nanowall surfaces.

Method 2: Tilt Sample in AFM. Most scans showed unexpected convex curving toward the top of walls, inaccurate wall slants, and other AFM interferences indicating untrustworthy roughness measurements. Only a few accurate scans could be used for sidewall roughness calculations, showing a 68 nm z-range and 10 nm RMS value on a 3 × 3 μm scan. The method was considered too unreliable for use without further improvements.

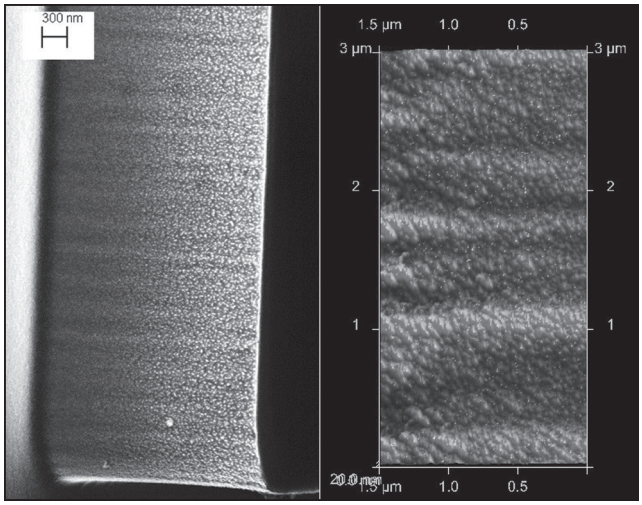

Figure 1: A comparison of SEM (left) and AFM (right) scans of the same structure using Method 3.

Wafer, by etch time	With polymer		After polymer strip	
	Z-Range (nm)	RMS (nm)	Z-Range (nm)	RMS (nm)
5 min #1	66	8	121	16
5 min #2	37	5.4	24	4.3
10 min	30	4.4	47	5

Figure 2: Average roughness of nanowall sides over 2-15 µm² area with horizontal resolution 3.5-47 nm [4].

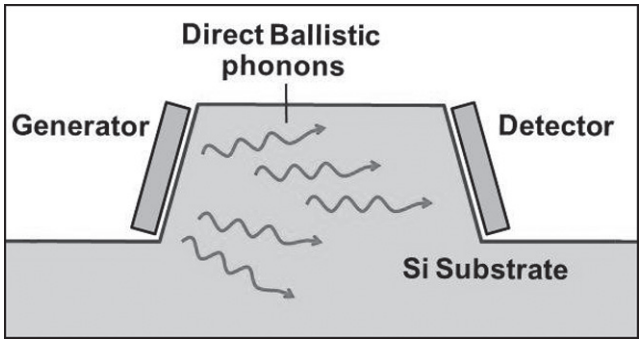

Figure 3: Model of the phonon spectrometer.

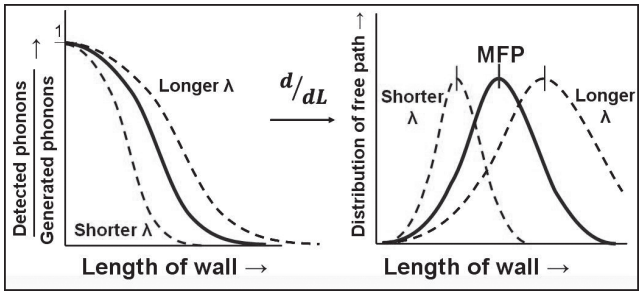

Figure 4: Schematic form of the expected relationship of phonon transmission versus nanowall length.

Method 3: Break and Tip Substrate. All samples tested yielded AFM scans that corresponded well to structures observed under SEM (see Figure 1). Walls parallel to cleave planes were most easily observable. The method was used to measure surface roughness of samples retaining a layer of polymer deposited during continuous passivation etch and after the polymer was stripped using the Gasonics Aura 1000, with results shown in Figure 2. The data was inconclusive as to the effects of the fabrication step but demonstrated that this method can be used reliably and quantifiably to analyze the effects of fabrication conditions.

Conclusions:

Method 1 was not viable for future use. **Method 2** was the least destructive and most applicable of all methods, but was too unreliable for immediate use, requiring further improvement. **Method 3** was the most destructive method in that an entire device would be cleaved in half for measurement, but at the same time was the most reliable and therefore the best method for immediate use.

Future Work:

The phonon spectrometer shown in Figure 3 will be used to find thermal conductivity through silicon nanowalls experimentally, and the relationship between conductivity and nanowall length will be established. The schematic form of the expected relationship and determination of mean free path (MFP) is shown in Figure 4. Fabrication processes will be varied to alter surface roughness and obtain the relationship between roughness and MFP, which will aid researchers in modeling thermal conductivity in devices.

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