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# Temperature Treatment on Silicon Nanowires for Reliability Studies

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*Abstract*— **Material reliability is among the crucial factors that impact material performances before device applications. In order to predict material reliability, accelerated aging study—a study to predict material shelf life when subjected to temperature, was performed on Silicon Nanowires. We investigated the effects of process conditions on the diameters and the quality of Si NWs using Atomic Force Microscopy. The experimental results revealed diameter of Si NWs has linear relationship with varying temperature. These results are of significant importance and will be a critical design consideration for the manufacture of Nanoelectromechanical systems involving Si NWs.**

## INTRODUCTION

Nanowires (NWs) are one of the basic building blocks for nanoelectromechanical systems (NEMs). Many different types of NWs exist including metallic NWs – Nickel (Ni), Platinum (Pt), Gold (Au); semiconducting – Indium Phosphide (InP), Silicon (Si), Gallium Nitride (GaN); insulating NWs –Si Dioxide ($SiO_2$) and Titanium Dioxide ($TiO_2$). Currently, there are tremendous interests in one-dimensional (1-D) nanostructures, such as nanowires and nanotubes, due to their potential to serve as critical building blocks for emerging nanotechnologies [1-5]. Of particular importance to 1-D nanostructures is the electrical transport through these "wires", since predictable and controllable conductance will be critical to many nanoscale electronics applications. One-dimensional nanostructures such as semiconductor nanowires are attractive as building blocks for the assembly of nanoelectronics and nanophotonics systems because they can function both as nanoscale devices and interconnects [6].

Silicon nanowires (Si NWs) represent a particularly attractive class of building blocks for nanoelectronics because their diameter and electronic properties can be controlled during synthesis in a predictable manner [7-9]. The ability to control the electronic properties has been utilized for the reproducible assembly of field-effect transistors (FETs) [10-12], logic gates [13] and sensors [14]. In addition, recent studies suggest that Si NWs FETs can exhibit transport characteristics that are comparable to or exceed the best planar devices fabricated by top-down approaches [11]. Research into Si NW's thermoelectric and electrochemical properties have also shown it to be ideal for use in thermal couples and battery anodes [15-16].

Carbon nanotubes had shown prominence in the field of one-dimensional nanostructures. However the size dependency of electrical properties and the inability to alter the electrical characteristics through doping have limited their usage. Si NWs on the other hand, with their ability to alter electrical properties offer flexibility to work with and thus intensely studied by many researchers and scholars. However, before taking full advantage of Si NWs, it is critical to understand its reliability under environmental conditions such as temperature and humidity. The understanding of Si NWs reliability will help facilitate the modeling of the mechanics for changes in physical and mechanical properties as the Si NWs related nanosystems age.

Accelerated aging is the study of material shelf life and is often performed in laboratory settings. It is the most adapted test to predict the life time when there is no scientific data available. In this test the material is subjected to excessive oxygen, temperatures, and sunlight in order to accelerate its actual aging [16-17]. The material properties such as the mechanical fatigue, load cycle intake, material stability are evaluated for the prediction of shelf life of the material. Though Europe has favored standard testing methods based on aging at elevated temperatures, slicing and scaling techniques have been the leading approach in North America [18]. Recently standard test methods in Canada have been adapted by many industries across the US, but these test methods are only defined for the macroscale components. As the test methods involve slicing the material [19-23] which is impossible at the micro- and nano- scale levels, these methods cannot be directly adapted for micro and nano scale materials.

Accelerated aging testing is based on a thermodynamic temperature coefficient formulated by Von't Hof which that states "*for every 10 degree C rise in temperature the rate of chemical reaction will double*." However, since this formulation is based on rate kinetics of a single chemical reaction, not on packages with various kinds of materials, the direct extrapolation of this theory to the aging of packaging materials must be used with caution.

In order to overcome the above shortcomings and to predict the reliability Si NWs, a new accelerated aging experiment was carefully designed and tested based on Taguchi's approach [24-30] for nanomaterials. Using this designed experimentation schemed, Si NWs reliability was studied by subjecting them to varying temperatures, and their dimensional changes (diameter) was recorded using the Atomic Force Microscope (AFM). Statistical analysis was performed on the data collected to enable reliability study and facilitate the modeling of the mechanics for changes in physical and mechanical properties as the Si NWs related systems age.

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## MATERIALS AND METHODS

Si Nanowires (>99%) and acetone (>97%) was purchased from Sigma Aldrich. Seed Solution: 40 mg of Si NWs was measured using an electronic balance with a resolution of 10 µg and suspended in 1 mL of acetone to make a seed concentration of 40 mg/mL. From the seed concentration, the final working concentration of 0.1 mg/mL was prepared by mixing 250 µL to 1 mL. In order to ensure a uniform dispersion, the seed solution was sonicated in a sonicator for 1 hour and then the final working concentration was prepared. The final working concentrations was again sonicated for 1 hour and then dispersed onto the glass slide. The acetone was evaporated using a hotplate. The Si NWs dispersed glass slides were then subjected to variable temperatures at a low (3%) humidity.

A Microclimate Environmental Chamber (Manufactured by Cincinnati Sub Zero model No. MCBH 1.3) was employed for the study. In the experiment the temperature was ramped between 22°C and 150°C with a step increment of 10°C for 30 minutes. The AFM was then utilized to obtain surface images of the treated Si NWs after returning to room temperature while kept in a dry box with a relative humidity of 3%. The changes in the dimensions of the Si NWs were then recorded.

## EXPERIMENTS: DIMENSIONAL ANALYSIS USING AFM

The initial diameters and lengths of the silicon nanowires before exposure to the environmental chamber were 40 nm and 1 – 20 µm respectively for 99% of the Si NWs. In order to accurately determine the diameter of the treated Si NWs, an Atomic Force Microscope (AFM) is employed. The AFM used in the experiment is the Agilent 5500-ILM highly sensitive and precise microscope. A diagram of the AFM probe and a diagram of the AFM set to Acoustic Tapping (AC) mode can be seen in figures 1 and 2 respectively.

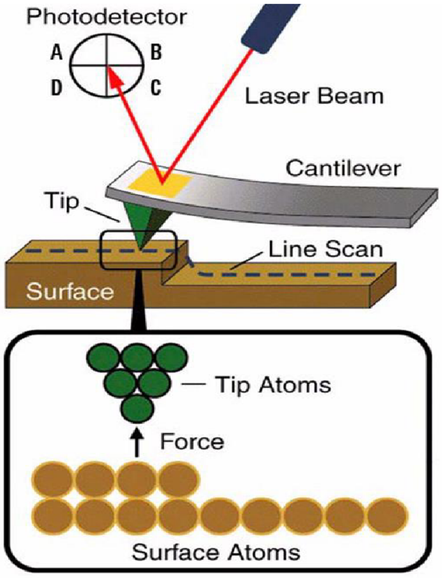


Fig. 1. AFM probe diagram showing detection of tip-surface interaction

The AFM probe utilized during imaging has a resonant frequency of 190 kHz and a spring constant ($F_c$) of 48 N/m. Prior to the experiments, the tip sensitivity was calculated. The sensitivity ($S$) of the tip is the ratio of deflection of cantilever to the applied amplitude. The tip was calibrated for sensitivity with respect to a mica surface and the tip sensitivity is 66.4 nm/V. A setpoint voltage ($V$) of 0.4 V was used for the accelerated aging study. Thus the applied tip force $F_{tip}$ was obtained as 1.275 µN using the equation below. The Si NWs samples were then loaded into the AFM for scanning and measurement of the diameter changes. During intermittent contact, the tip is brought close to the sample so that it lightly contacts the surface at the bottom of its travel, causing the oscillation amplitude to drop. Hence, the influence of the cantilever tip during the dimension measurement can be neglected since it will not change the shape in this mode of contact.

$$F_{tip} = S \times F_C \times V$$

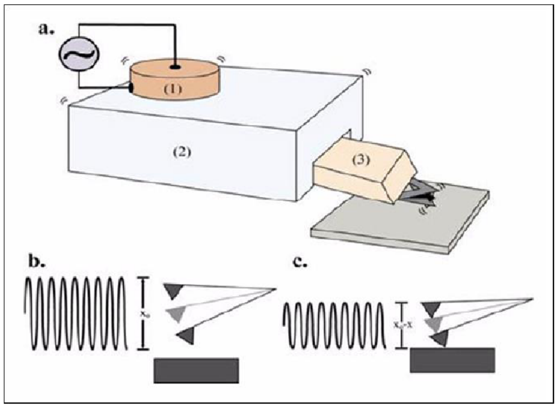


Fig. 2. AFM probe motion under Acoustic AC Mode: a. (1) AC applied to the nose cone; (2) the base body of the cantilever beam; (3) the cantilever bean with its tip; b. & c. the cantilever driven to oscillate in sinusoidal motion.

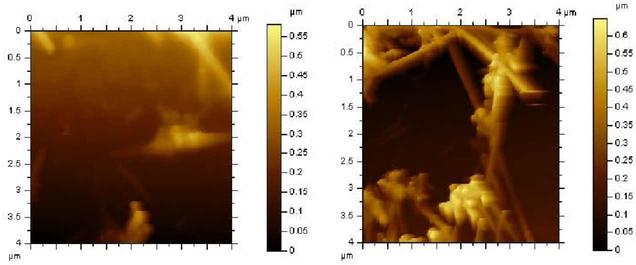


Fig. 3. Left. Scan of Si NWs on glass prior to treatment (22° C was room temp.) Right. Scan of Si NWs after treatment at 150° C.

A scanner (max scan size 9 µm × 9 µm) with Aluminum (Al) coated tip was used for scanning the samples. At first, in order to locate the Si NWs, a large scan area of 8 µm × 8 µm is scanned. The scan areas of the Si NWs before and after environmental chamber treatment are shown in Fig. 3. As depicted in Fig. 3, nanowires were dispersed over the glass plane and once the nanowires were located, a cross section was drawn across in order to obtain a two dimensional graph of the nanowire. A cross section from the AFM is shown in Fig. 4. The dimensional data can then be obtained from the graph. The peak of the curve from the baseline is the diameter of the nanowires.

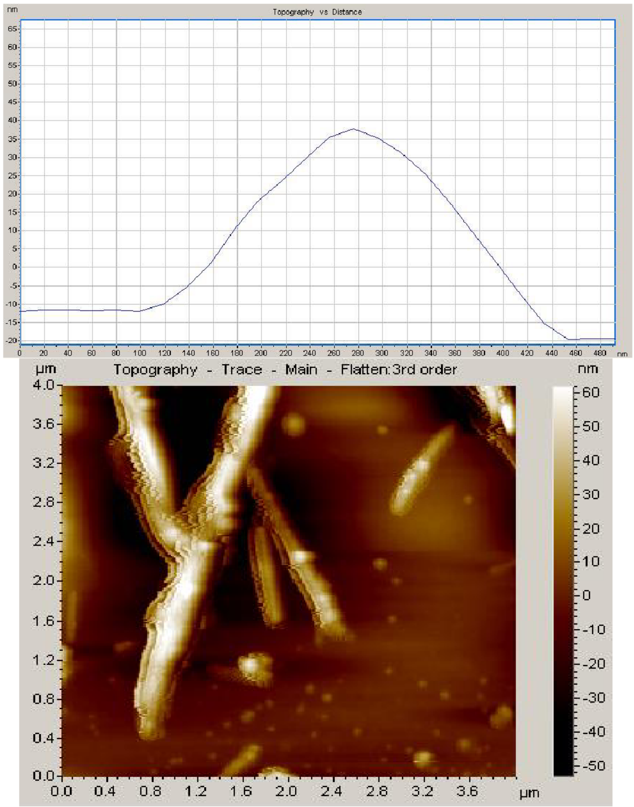


Fig. 4. Top: Cross section of Si NW after treatment at 80° C showing a diameter of around 50nm. Bottom: Corresponding scan (4µm x 4µm) of Si NWs after treatment at 80° C

## RESULTS AND DISCUSSION

Figure 5 shows the data distribution generated from the 2D images after 22° C, 50° C, 100° C, and 150° C treatments. The average diameters for 10 nanowires were plotted at each of the temperature intervals. It appears that the diameter of the Si NWs have a linear correlation to temperature as seen in Figure 6. Recorded data was tabulated and the standard deviation for each set of measurements was calculated. Standard deviation for measurements of nanowire diameter varied between 2.64nm and 5.536nm as shown in Table I.

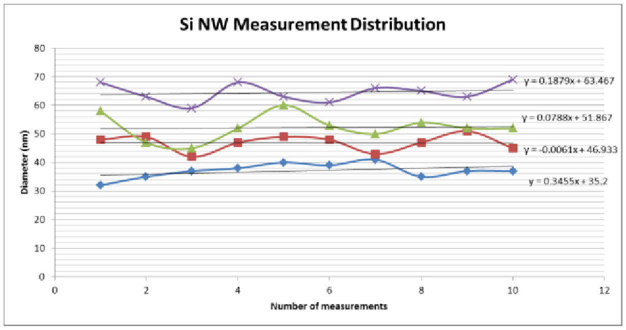
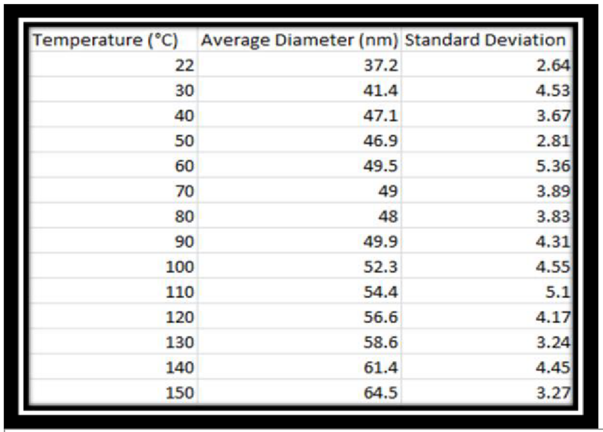


Fig. 5. Analysis of diameter measurements taken at 22° C (blue), 50° C (red), 100° C (green) and 150° C (purple)

TABLE I
AVERAGE DIMENSIONAL DATA COLLECTED FROM AFM AFTER TREATMENT AT DIFFERENT TEMPERATURES



| Temperature (°C) | Average Diameter (nm) | Standard Deviation |
|---|---|---|
| 22 | 37.2 | 2.64 |
| 30 | 41.4 | 4.53 |
| 40 | 47.1 | 3.67 |
| 50 | 46.9 | 2.81 |
| 60 | 49.5 | 5.36 |
| 70 | 49 | 3.89 |
| 80 | 48 | 3.83 |
| 90 | 49.9 | 4.31 |
| 100 | 52.3 | 4.55 |
| 110 | 54.4 | 5.1 |
| 120 | 56.6 | 4.17 |
| 130 | 58.6 | 3.24 |
| 140 | 61.4 | 4.45 |
| 150 | 64.5 | 3.27 |

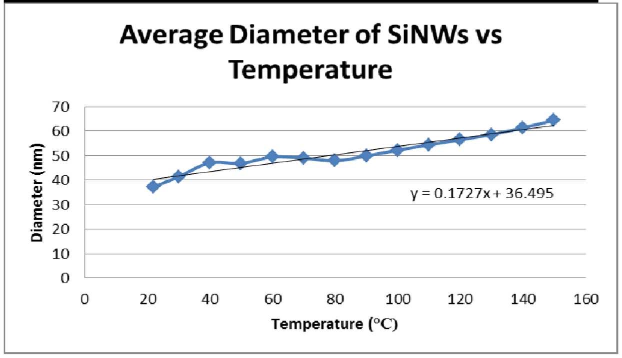


Fig. 6. Average diameter relative to temperature the wires were treated at.emperatures.

## CONCLUSION

From the present study, the reliability of the Si NWs can be studies under the conditions of temperature. The current results confirm the need to make the necessary design considerations when Si NWs based devices are being researched in the conditions of variable temperatures as the material dimensions at the nanoscale have significant impact on the material's properties. The present study also concludes that the Si NWs diameter had a linear relationship with respect to temperatures in the range of 22 °C – 150 °C. TEM imaging measurements of a Si NWs aged at 50°C showed that the oxide layer thickness had increased to 14.22 ± 1.12 nm, which is a significant. Further research is needed to model the mechanics for changes in physical and mechanical properties as the systems age. Additional accelerated aging tests also need to be done on Si NWs while under acidic pH levels typical of a lithium-ion battery environment as well as low temperatures typical of space conditions.

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