Polysilicon Nano-wires Based Nano-device on Silicon Chip: Fabrication and its Application

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ABSTRACT

5x5 arrays of six nano-transistor devices were fabricated on p-type silicon/silicon oxide (200nm) wafers with resistivity 1-100 Ω-cm. 50nm of doped polysilicon were deposited and annealed to make nanowires. All 50nm nanowires were made with electron beam lithography (EBL) and unwanted polysilicon were etched with chlorine plasma. These nanowires were connected with 250nm wide gold source and drain electrode with 150nm separation. Device characteristics were tested with electrochemical impedance spectroscopy (EIS) using potentiostatic Bode, Nyquist and Schottky-spectra. The device was also tested with varying gate voltage using a digital voltammeter. The device was stable in the voltage range 0.0-0.5V and able to pass 100 μA current smoothly. Most devices were found to be as field effect transistor and other devices were like diodes. Finally, various capture molecules for specific bio-detection were immobilized on a nano-device sensing surface by using a simple adsorption approach as well as covalent linkage chemistry, verified by a using fluorescent microscopic method. The modified nano-biosensor was incubated with ~300nL biological sample containing 1 nM to 10 fM target molecules and measured with EIS. The detection sensitivity of nano-FET biosensor was found in the range of 40fM-100fM with 50-70% of reproducibility.

Keywords: nano-fabrication, polysilicon nanowires, electrochemical impedance, nano biosensor.

1 INTRODUCTION

Polysilicon nanowires in sensing application offer many opportunities for the construction of a biosensor nano-device. Their robust structure, long term stability and reliability, and easy fabrication have attracted much attention from the research community. Various researchers have shown working nanowire devices, such as nanoscale field-effect transistors (FETs), diodes and other nano-device [1-6]. In all of these reports, nanowires were often removed or displaced from the substrate on which nanowire were made and placed into solution. In our fabrication, polysilicon nanowire based nano-device was protected with very thin polyimide layer, which shows great promise, very sensitive, fast and simple bio-detection with state of art immobilization of capture probe on polyimide surface. These devices are very useful and can apply to any bio-detection with changing the capture probe on a sensing surface according to their specific target.

2 METHODS

2.1 Device Fabrication

Doped polysilicon nano-wire devices were fabricated at the Cornell Nanoscale Facility (CNF), Cornell University, Ithaca NY. A four-inch MOS clean p-type silicon /silicon dioxide wafers were first deposit 50nm of a p+ type polysilicon film and annealed. The key opening layer for alignment marks were then patterned and deep etched in plasma. Nanowires were created by spinning resist SPR 220-3.0 i-line at 4000rpm for 60 sec, baked at 115ºC for 90 sec. All wafers were exposed using GCA AS200 auto-stepper and baked again at 115ºC for 90 sec. These wafers were developed in AZ 300 MIF (tetramethylammonium hydroxide), air dried and inspected under microscope. These wafers were then deep etch in CF_4 plasma. After etch, unwanted resist was removed by placing wafers in hot resist bath for 20 min. All the striped and clean wafers were spin coated with 6% XR-1541 HSQ (hydrogen silsesquioxane resin) e-beam resist at 6000 rpm for 60 sec and baked at 170ºC for 2 min. The nanowires were patterned using leica VB6 e-beam. Exposed wafers were post baked again at 170ºC for 2 min. These baked wafers were developed in microposit MF-321 developer (tetramethylammonium hydroxide & surfactants), rinsed with DI water and dried under nitrogen. The patterned nanowires wafers were then etched in chlorine plasma. The above e-beam the lithography procedure was repeated again

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to get the small gold connection pad on each end of the nanowires. Proximity correction was made before exposure on the e-beam to the bond pad pattern. After proximity correction, the wafers were exposed on the e-beam. These wafers were post baked at 170ºC for 2 min and developed. All wafers were then descum in oxygen plasma for 90 sec to remove any resist residue. After descum the titanium (10 nm) and gold (50nm) were deposited on these wafers using a CVC SC4500 E-gun evaporator. After metal film evaporation, the wafers were left overnight in a microposit remover 1165 (N-methyl-2-pyrrolidine) to lift-off unwanted gold. The wafers were then spun with hexamethyldisilazane, (HDMS) primer and followed by OiR 620-7i resist at 4000 rpm for 60 sec. These wafers were prebaked at 90ºC for 60 sec. After resist bake, the patterns were created with an auto-stepper for 0.5 sec UV exposure. Exposed wafers were post baked at 90ºC for 60 sec and developed in automated developer unit using AZ 300 MIF developer for 2 min and descum in O_2 plasma The CVC SC4500 E-gun evaporator via E-beam was used again to deposit titanium (10 nm) and gold (50 nm) films and left 12 hrs in 1165 to lift off unwanted gold. Photoneece® PWDC-1000 positive tone polyimide was spun on the patterned glass wafer at 6000 rpm for 90 sec and baked for 1 min at 110ºC. The auto-stepper was used again to expose the polyamide for 0.5s to open contact pads on the device. The wafers were developed in MF 321 developer, cleaned, and descum. The final cure of PWDC 1000 film on the device was done in a YES 450PB polyimide oven for 8 hr. After the polyimide bake the wafer was inspected under a microscope. The outline of fabrication depicted in Figure 1.

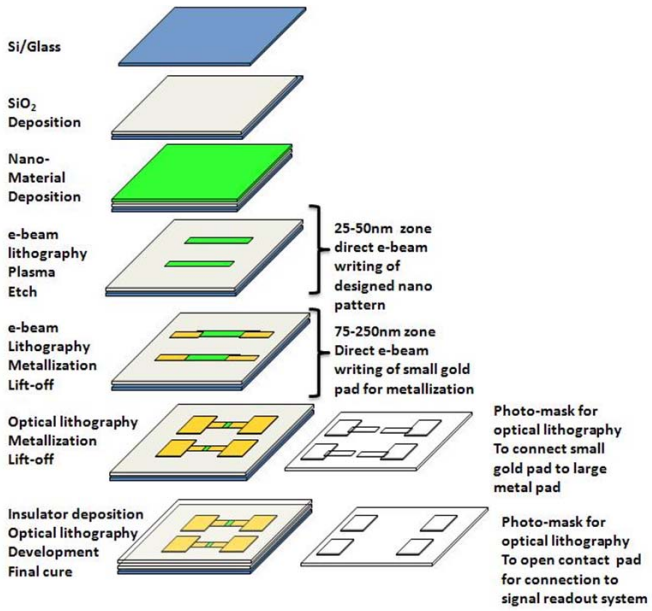

Figure 1. Schematic of fabrication steps used in polysilicon nanodevice fabrication.

2.2 Imaging of Nano-Devices

Scanning electron microscopy (SEM) is very critical for the analysis of nanoscale materials and structures. Zeiss Ultra 55 microscope optimized for high resolution imaging was used to scan the pattern polysilicon nanowire operating at beam energy 1.5 kV (see Figure 2.). The microscopic images of device were taken with an Olympus BX60 UV microscope (see Figure 3.)

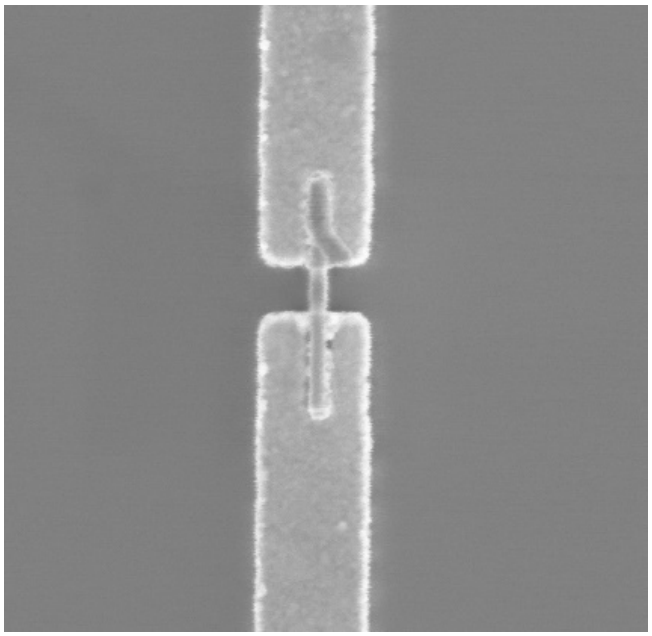

Figure 2. 100 KX SEM image of fabricated polysilicon nanowires device showing 50nm width wire connected with 2 gold pad with 150nm gap. Aperture size was 30 micron with 3nm pixel size. 2 signal Inlens detector was used to scan this image.

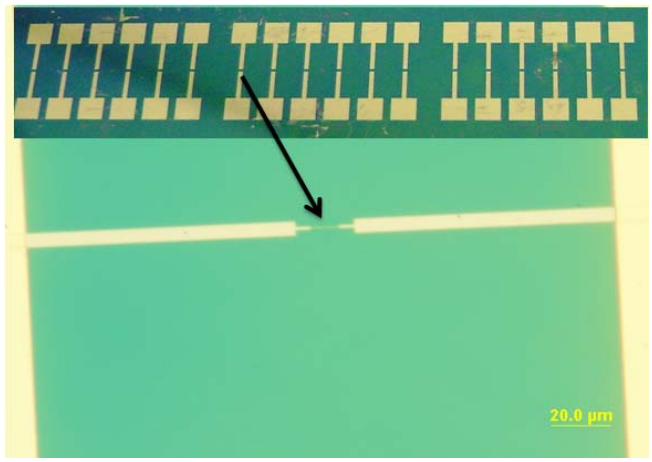

Figure 3. Camera and 20 X microscopic image of fabricated gold nano-device device. Microscopic image was taken under polarized light.

2.3 Electrical Characterization

Electrochemical impedance spectroscopy (EIS) was used used to characterize the device nature from 0.02 Hz-100 kHz frequency range with using a Ref600 electrochemical system from Gamry Instruments USA. 0.25 µl of test biological samples were loaded onto the sensing surface of nano-device at room temperature for 15 min, washed and EIS spectra was recorded. The EIS Bode and Nyquist plots were analyzed with Gamry's Echem software. I-V measurements of three terminal polysilicon nanowire devices were taken with the Keithley Model 2602 source meter. The script was designed as per nano-FET test data finding from the EIS measurements. The data was plotted using MATLAB R2007b.

3 RESULTS AND DISCUSSION

The 50nm polysilicon nanowires have been patterned using e-beam lithography with precisely RIE using chlorine plasma. These nanowires were connected with 250nm gold pads with 150nm gap between them. These 250nm wide gold pads were made using e-beam. To obtain uniform exposure proximity corrections were made before exposing to e-beam. Proximity correction has been found to be useful for two general classes of patterns. The first is patterns with multiple feature sizes, which require different exposure doses to be used for each size. An example is shown in Figure 4. Different e-beam doses were assigned automatically to the narrow and wide features to obtain developed structures with high fidelity to the designed pattern. 250-600nm polyimide insulating layer was patterned on top of the nanowires using optical lithography. All devices were tested under varying reference voltage from 0-350mV and AC rms sweep at 50mV. Field effect behavior of the nano-devices were clearly observed on EIS bode plot (see Figure 5). In the lower frequency zone all devices showed to decrease in modified impedance, which in fact is an indication of transistor behavior with increasing bias voltage from the third terminal. The field effect behavior of the polysilicon nanowires with different doping levels was also investigated. I-V characteristics of some p-type polysilicon nano-device were also shown changes that corresponds to gate voltage (see Figure 6). In this test, increase in current was observed with backward gate voltage (-0.3V) on forward Vds. That suggests device sensitiveness to small applied bias voltage. More I-V measurements are necessary to analyze complete electrical behavior of these polysilicon nanowires in the device. Nyquist EIS of pure biological protein sample [6] from 1nM to 1fM were used to demonstrate detection on this fabricated nano-device (see Figure 7). A clear change in impedance was observed in nyquist semicircle with change in protein conc. demonstrating the device sensitivity to sense any change on its surface due to these protein samples.

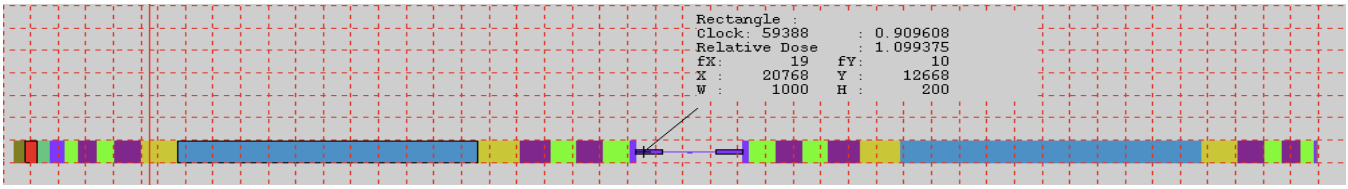

Figure 4. Proximity correction on nano-device gold contact pad is shown. Dotted squares are 5 µm in size.

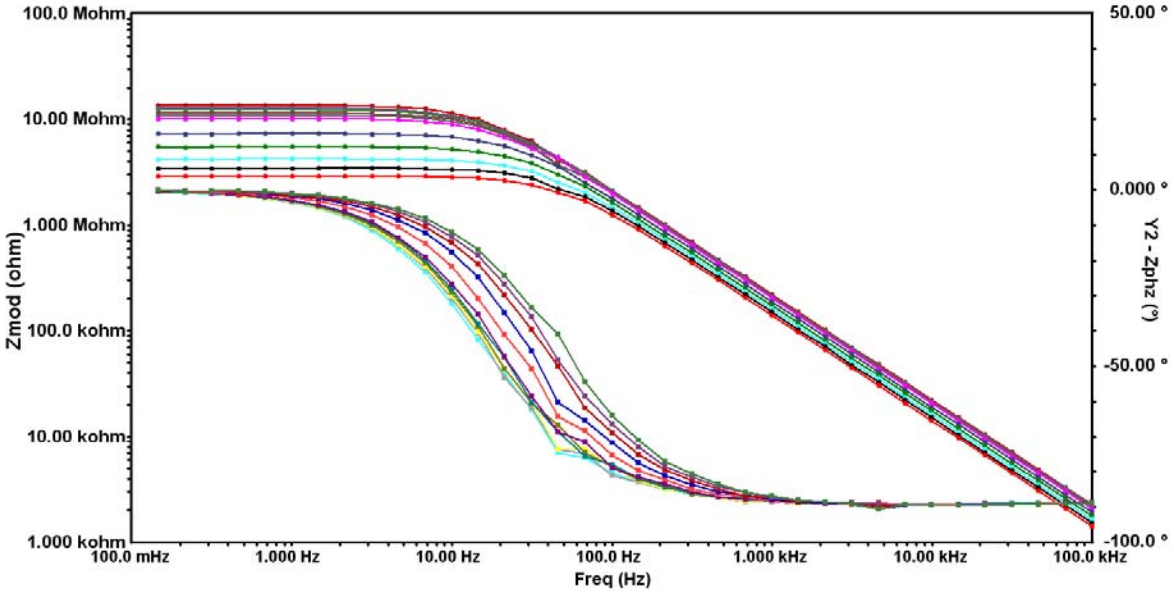

Figure 5. EIS bode plot of 50nm polysilicon nanowires device showing decrease in modified impedance with increase in bias voltage.

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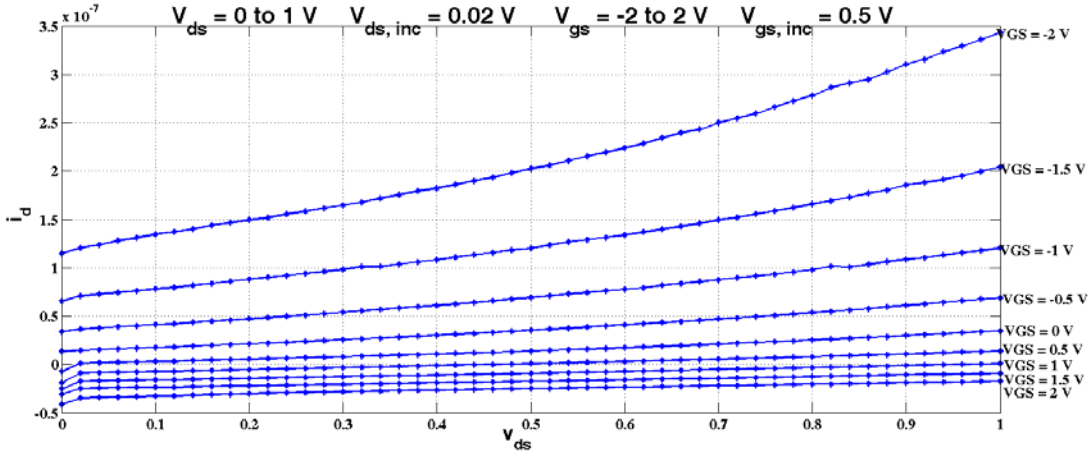

Figure 6. I-V Plot of nano-device at -2V to 2V gate voltage with 0V to 1V from drain to source terminal. The curve is clearly showing transistor type behavior with p-type channel on the device

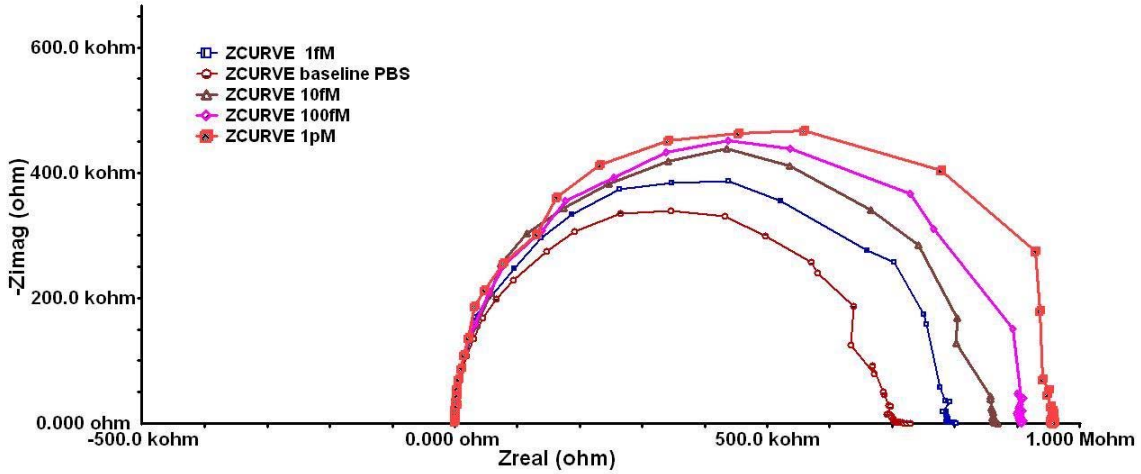

Figure 7. EIS nyquist plot of nano-device showing changes in real and imaginary impedance with increasing conc. of protein samples from 1pM -1fM concentration.

4 CONCLUSION

We successfully demonstrated the sensitive nature of polysilicon nano-device due to the change in the device surface with applied bias or with biological samples in fM range concentration. The device has potential application in detection of biological samples. Various other characterization, surface modification and optimization of other experimental conditions for future integration with CMOS chip is underway.

5 ACKNOWLEDGEMENTS

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REFERENCES

[1] Y. Cui and C.M. Lieber: Science **291**, 851, 2001.
[2] X.F. Duan, Y. Huang, Y. Cui, J.F. Wang and C.M. Lieber: Nature **409**, 66, 2001.
[3] J.F. Wang, M.S. Gudiksen, X.F. Duan, Y. Cui and C.M. Lieber: Science **293**, 1455, .2001.
[4] A.M. Morales and C.M. Lieber: Science **279**, 208, 1998.
[5] Y. Cui, X. Duan, J. Hu and C.M. Lieber: J. Phys. Chem. B **104**, 5213, 2001.
[6] A.K. Pant, S. P. Murarka, C. Shepard, and W. Lanford, J. Appl. Phys. **72**, 1833, 1992
[7] W. C. Maki, N. N. Mishra, E. G. Cameron, B. Filanoski, S. K. Rastogi, G. K. Maki, Biosens. & Bioelectron., 23, 780–787, 2008.

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