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Design, Fabrication and Characterization of silicon Nanostructures for Lead (Pb⁺) ion detection

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Abstract. Silicon nanostructure were prepared using cheap and in-house technique developed in INEE. Amino-functionalized Si nanowires were tested against heavy metal lead (Pb). The device was fabricated via dry oxide etching approach with control oxygen flow rate in oxidation furnace, network of uniform Si nanowires was successfully fabricated. The device was functionalized by (3-aminopropyl) triethoxysilane (APTES) to save as a sensor to heavy metal. Due to the silicon electrochemical response toward heavy metal ions, linear response to three different sources of water were observed. The results indicated, Pb can be detected with high precision. Furthermore, confirmation was demonstrated using atomic absorption spectroscopy instrument (AAS) to determine the level lead content in each water source. Three sources of water were tested: Tap water (H₂O), River (H₂O), DI (H₂O) and 0.0859mg/L, 0.0929mg/L, 0.023 mg/L with ~ 52pA, 60pA and 70pA current response respectively. Thus, with this high capability to discriminate water samples, the sensor potential to be employed for an effective heavy metals detection and can further be extended for large sensor network in water treatment plant.

1. Introduction

In the recent time, the silicon based devices in an nanoscales have attracted attention of many research communities this is because the material has shown potential prospect to broad applications in different filed, this include electronics related devices research and manufacturing, photonics related applications, the device equally shown considerable potentials in renewable energy to biomedical sensing [1,2]. Significant efforts have been employed by many researchers to used in application such as chemicals and in quest of this, new silicon nanostructures have been proposed and fabricated, this included quantum dots silicon devices device, silicon nanowires or silicon nanopores [2,3]. The silicon nanostructures such Nano-IDE, in particular, have attracted interest of the many researchers due to its good dimensional properties



which of good to its electrical and mechanical properties [1, 3]. Moreover, The properties of silicon based devices such as indirect band gap, silicon based device can easily be explored in creating active material for functional electronics device that could use in specific application [1, 4, 5]. Decreasing dimensions to smaller pieces with 0.1 μm (100 nm) average length will not only influence the band gap, but further decrease of dimensions to even smaller pieces with average dimensions less than 10 nm will change the band gap and leads to appearance of new properties such as visible light or enough catalytic activity for a specific reaction [5,6]. These new properties are related to the “size effect,” and 100 nm is approximately the border: nanostructures with dimensions lower than this amount will be affected by the size effect [7]. The reasons for the different properties of materials on the nanometric scale or their nanometric size effect have created many industrial applications of nanostructures [8]. Electrochemical sensing of heavy metal ions with inorganic, organic and bio-materials are currently occupying the mind every researching working in this field. Cui et al, have hypothesized in study that electrochemical sensor has short analytical time, low power cost, high sensitivity and easy adaptability for in-situ measurement [9]. When they were summarizing and discussing recent achievements in electrochemical sensing of heavy metal ions within organic and bio-materials using modified electrodes [10]. From their findings, they conclude that, the electrochemical measurements are the most viable method of analyzing chemical because they accurate, cheaper, simpler and easier for handheld device which could promote real time detection [9,10]. However, the device suffers a very big draw back because most researchers with these devices do only a proof-of-concept in detection of its targets [11]. They further found that, this particular sensor can be used not only for environmental monitoring but also in the clinical, safety and security fields (Cui et al, 2015). Detection of chemical pollutants in water using gold nanoparticles as sensors: This research currently occupying the mind many researchers in this area [12] have hypothesized their study that Electrochemical sensor has short analytical time, low power cost, high sensitivity and easy adaptability for in-situ measurement [13]. When they were summarizing and discussing recent achievements in Detection of chemical pollutants in water using gold nanoparticles as sensors [14]. From their findings, they conclude that, silicon-based sensors provide promising approaches for selective and sensitive analyses, and have advantages over traditional instrumental analyses, as they are usually compatible with portable devices that can be deployed in field for onsite sample screening [15]. With the advance of microfluidic sample handling units, this kind of portable device can be developed for multiplexed, quantitative and rapid analysis, and can measure unprocessed field-collected water samples with a high sensitivity. High-throughput and ultrasensitive detection technology, based on nanowires, offer effective screening methods for many environmental analyses [16-25]. However, it should be noted that a number of important issues still need to be addressed before It can become real tools for environmental monitoring [26-38]. Methods of manufacturing reliable production silicon device based a large scale, with good clone, still lacks, Especially for anisotropic particles form [39-49]. Thus, this research proposed the in-house preparation of highly though put approach, fabrication, characterization and surface modification of silicon based electrochemical sensor that could detect lead ion in water.

2. Material and methods

100mm 4inch wafer, p-type silicon material was, prepared using R1 and R2 process, prior to pattern transfer, 4 wires were designed using AutoCAD and transferred to chrome mask for subsequent pattern transfer. Positive resist was prepared and spin coated using speed controllable spin coater, the coating speeds were increase from 0rpm to 3000rpm by incremental of 500rpm, this is done in order to achieve uniform coating. The resist coated samples (wafers) were place on hot plate at 110⁰C temperature for 15 minutes. The samples (wafers) were taken on the hot plate and cooled for 10 minutes. With this, the samples were ready for alignment and exposure. Via mask aligner, the samples were arranged and exposed to UV light for 60minutes. Due to the photo resist sensitive to the UV light, patterns were created and the pattern were fully revealed after washing away with resist developer. In this case, the samples were immersed in the developer for 5 minutes and removed and washed using DI (H₂O) and Hard

bake using the same hot plate at slightly different temperatures, the temperature used for the hard is 90°C . This is done in order to create link with resist molecule with silicon to avoid stripping during device pattern creation and subsequent pattern trimming. With this, the sample was ready for pattern creation and subsequent residual resist removal. The silicon micro wires were first created via reaction ion etcher. To do this, the sample were arranged in the etcher chamber and set all the required gasses to their desired level, the machine was on and kept for 5 minutes and gases were on and run 2 minutes for etching and another to minutes for resist removal. These were both done within this same machine. The samples were removed and the pattern were measured both radial and longitudinal dimensions, with this, the device ready for trimming. The trimming process, was done in two steps, first, initial trimming was conducted via oxidation furnace using dry etching process and lastly the, it then trimmed nano size via the reactive ion etcher. Thus, smooth and successful nanowires were fabricated and surface modified using APTES for heavy metals detection. After the fabrication its followed by surface modification where the silicon nanowire functionalized using 3-Aminopropyl Triethoxysilane (APTES) to enhance nanowire electrochemical activities and the nanowires were immersed in 2% APTES in water. $1.5\mu\text{l}$ APTES (0.01M) was dropped on an active area of silicon nanowire. The device was drying in dry cabinet for 30 minutes until it clearly dry. After that, the device were ready for electrical measurement

3. Results and Discussion

The morphological observation and nanostructures sizes were observed and prior the measurement process, samples were prepared, series of investigated were conducted using scanning electronic microscopy (SEM) and field emission scanning electron microscopy (FESEM). FTIR spectrometer. EDX, FTIR and XRD were employed in order precise understanding of materials elemental and material composition. The silicon nanowire was fabricated using typical in-house, pattern formation and dry etching approach, Si nanowires have been successfully fabricated figure 1, the device was fabricated with minimum energy effort as does not required two many steps to achieved nanowire. This can be observed in Figure 1. The Si nanowires were highly uniform figure 1b. After patterning using the mask aligner and etching with RIE for around 5mins, the size of Si nanowires around 100nm from their cross-sectional view.

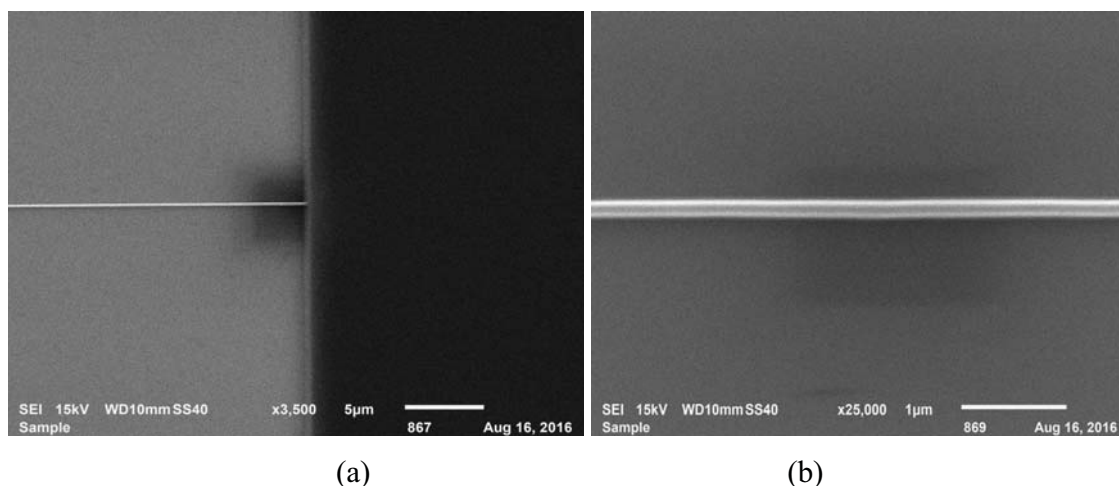


Figure 1. SEM observation for the nanowires image with 15kV acceleration range and 3500x magnification range

Figure 2, show the SEM images of the fabricated nanowires full length. The nanowire were uniformly and highly straight through out the nanostructure. From highly magnified SEM image shown in Fig. 2, it can be further confirmed that it the structure is approximately 100nm.

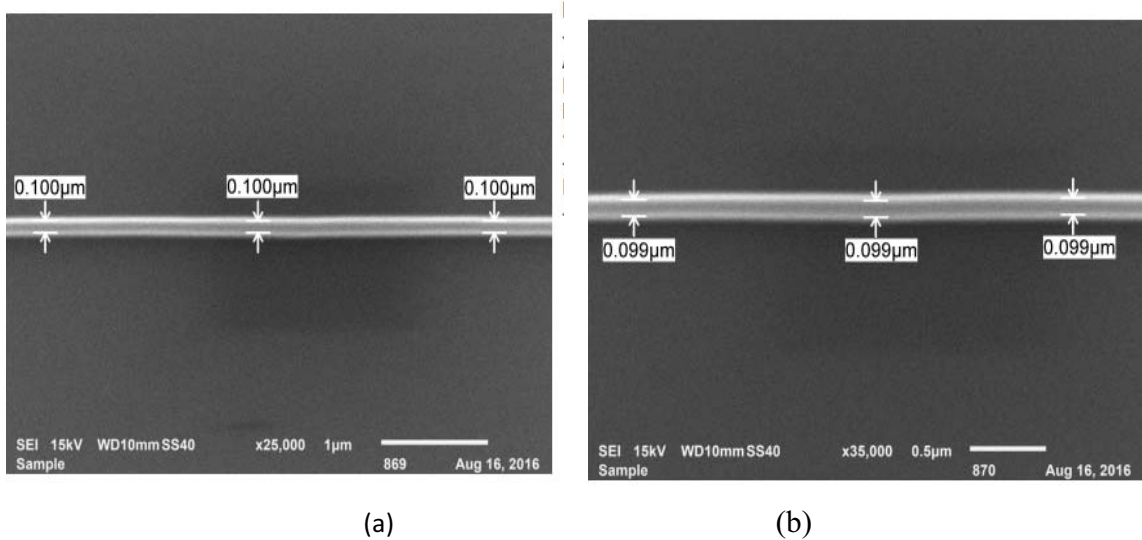


Figure 2. The observation of the nanowires SEM monograph

The silicon nanowires were fabricated as explained above by a simple, cheap and yet reproducible . figure 2a above the devices are entirely composed of silicon material and nanowires with lengths up to several thousand of micrometres. The typical diameters shown in both SEM AND FESEM of the nanowires are in the range of 90-00 nm figure 2b, this will allow high surface-to-volume ratios.

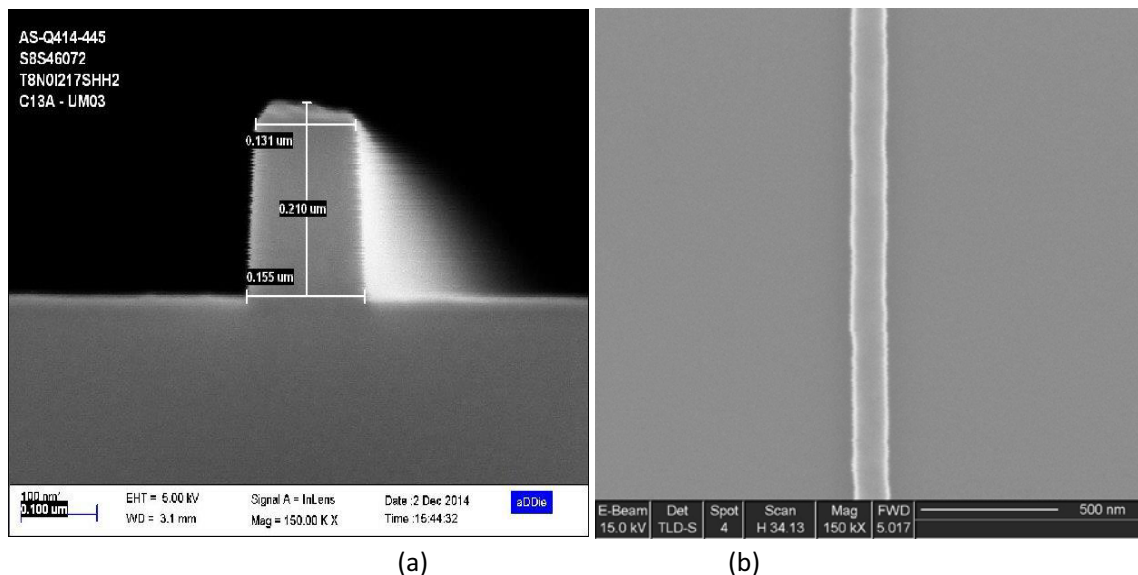


Figure 3. Nanowire crossection few with FESEM

The figure 3 results obtained from the FESEM also indicated, the profile of the the fabricated nanoires are straight and uniform with total surace intergrity with ~ 200nm height and 100nm width

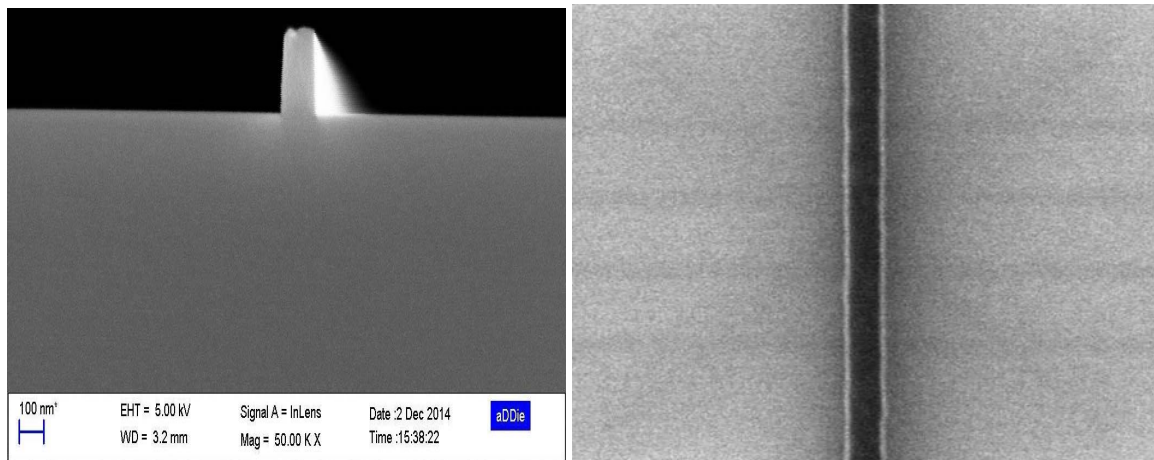
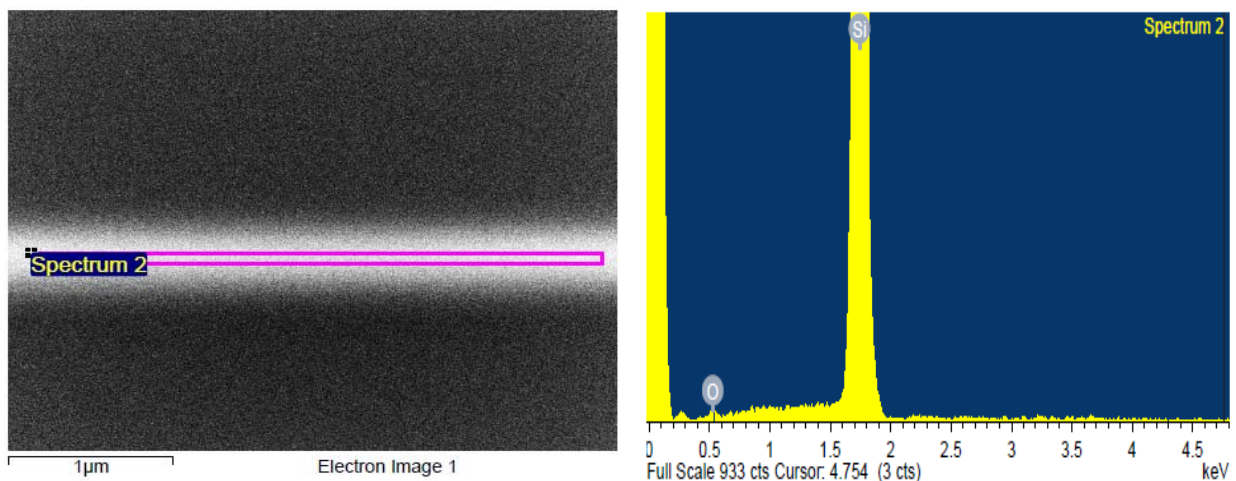


Figure 4. The cross section view based of field emission scanning electron microscope showing the fabricated nanowires

Moreover, based on the above figure 4, the nanowire is more or less rectangular with cylindrical curved ends, however, the original wire dimension after the pattern was between $1\mu\text{m}$ and height was before the oxidation etched was about $10\mu\text{m}$. After the thermal oxidation with removal from RIE etching of the layer at 1000°C for 30 minutes, and process consumed part silicon and some thickness converted to SiO_2 , this has led for the microwire to reduce to height of about 200 nm and with of 100 nm figure 4a. Thus, a pattern-size reduction by about more than $10\times$ times has been achieved. The process produce uniform and straight wire figure 4b. The precise control of nanostructure dimensions is a crucial point for a reproducible fabrication and for good electrical behaviour.



(a)

(b)

Figure 5. Show the Energy Dispersive X-ray (EDX) Spectroscopy which confirms the presence of Si and O on the nanowire.

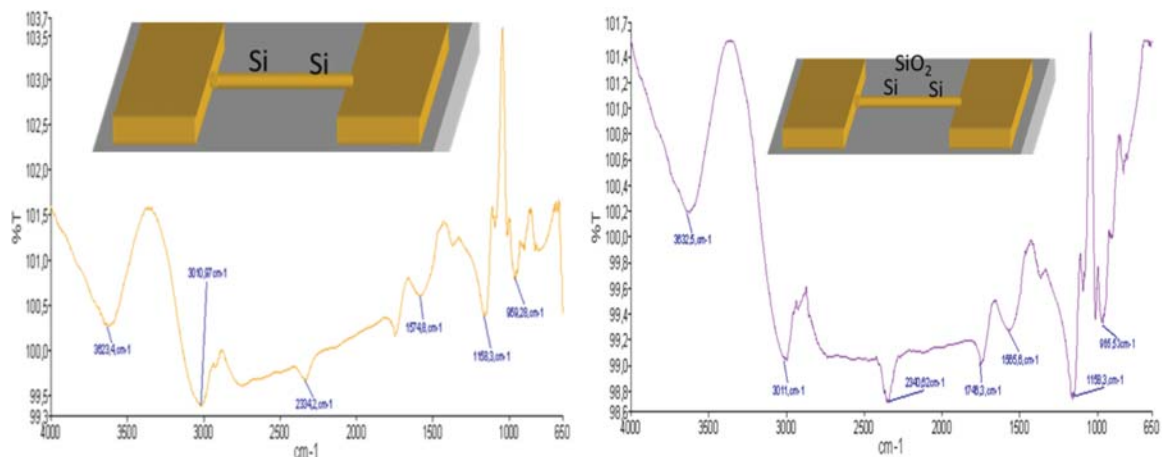


Figure 6. FTIR spectra of barred silicon nanowire and oxide functionalized Si Nanowires

The barred silicon have ability to response to the heavy metal ion, however, in order to enhance the response and complete absorption of ion, the silicon nanowire was surface treated, this will allow the Si nanowires toward to sensitive heavy metal ion. The present of the amino ($-NH_2$) in surface modification agent (APTES), functionalization process was conducted. The process was first started with barred wire was observed with FTIR as shown in figure above, Their schematic processes for the oxide deposition was presented in figure above are.. In Figure above the FT-IR spectrums barred silicon and oxide deposited device, the confirmed the EDX spectra explained prior this section.

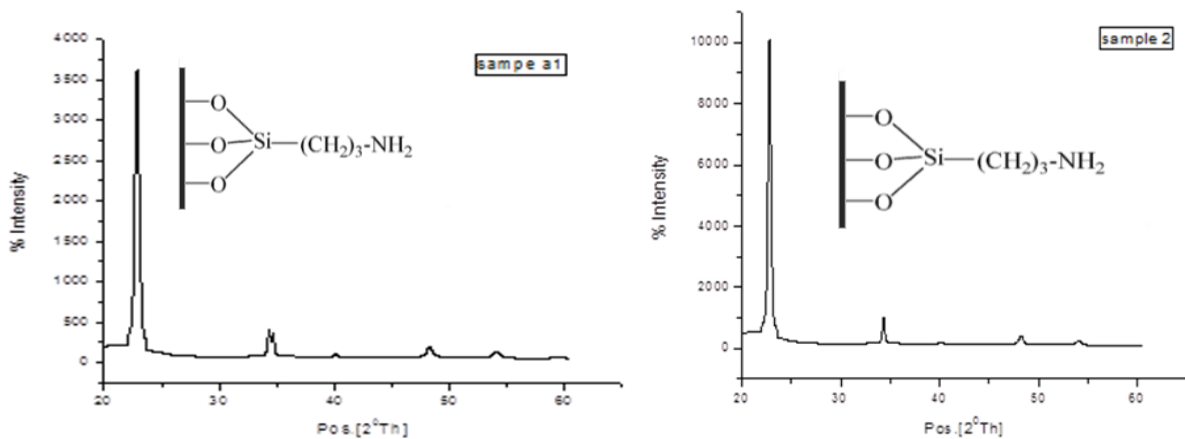


Figure 7. Aptes spectra

After the oxide deposition, aptes was dropped without further treatment, from the figure above in both samples it can be observed two major peaks one in each sample where the band with 3500 and 10000 % intensity with 20 to 30° and another minor peaks appears with the range of 30 to 40°, this minor peak within thin this band is ascribed to vibrating band of amino group of the aptes on surface of $-silicon$ nano wire NH_2 , For the other element such CH-R component of the aptes on-Si nanowires produce lower peaks, this might be due lack enough chemical activities and fully anchored limiting vibrations tendency. However, in amino $-NH_2$

groups. On Si nanowires, can be obviously seen have reveal vibration characteristic that have tendency to participant during heavy metal detection process, the CH-R component is indeed weak, however, it produced some vibration tendency that could enhance some reaction activities. Moreover, it can clearly seen and that can be concluded that aptes is exist on Si nanowires and are successfully grafted with functional groups of -NH₂. From the EDX, spectra, the above results of weak peaks also can be ascribed to present of the other element such oxide of silicon are present and a stiff silicon peak besides the peaks of oxygen is appeared for the Si nanowires due to the formation of a very thin film of SiO₂. Other peaks of very low intensity as moving from left to right arises as a result of residual of gasses element during the etched process in fabricating the nano structures.

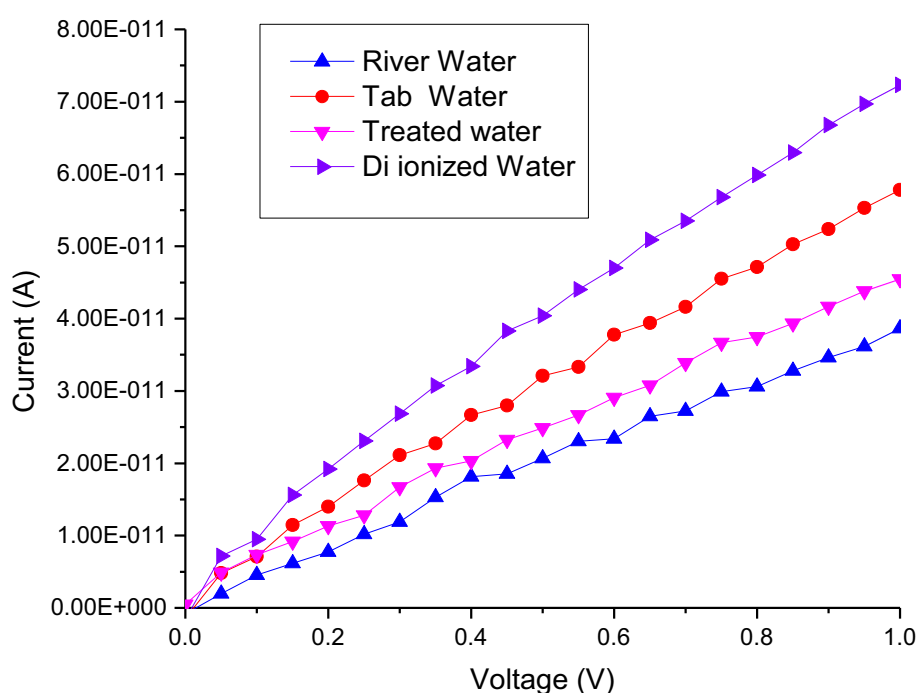


Figure 8. SiNW response to various water collected from 4 different sources

The device designed as sensor, it current response it determines its ability to response to other electrochemical activities which its response defined as the ratio of voltage to resistance. The voltages were swept from 0V to 1V using semiconductor analyser with input signal (0 V D.C Offset). Reference plane is full calibrated to reference plane and probing station was fully vacuum. Below is typical nanowire measurement at 100 nm. Primary current model for the three target analytes source of H₂O value shown in the graph for tab (H₂O), river (H₂O) and lake (H₂O with 0.0859mg/L, 0.0929mg/L, 0.1104 mg/L with ~ 52pA, 60pA and 70pA current response respectively. As shown in figure above, the response by barred nanowire, deionized water, lower than other three source of water, that might lack of the electrochemical activities contributed by the two afore mentioned samples. The factors that ultimately determine the present of the ions in the heavy metals. The river and the lake water normally full of heavy metals cations, which include Pb²⁺, The existing of this metals ions are reveal by the sensors response from the figure above indicating the, ability of the sensor to response, different ions and when observed closely, even the

treated water have still contain certain ion present and this is due to the alkaline and alkaline earth metal cations commonly, possess weaker coordination bonding with functional amino groups, whereas heavy metal ions exhibit a much stronger bonding with amino ligands, this has resulted in a remarkable selectivity, the claimed equally reported (Wei et al, 2014). The APTES amino group (NH_2) are receiving serious attention in the enhancing sensors to recognize heavy metals ions due to its specific and selective recognition of heavy metals ions, the interaction between heavy metals amino group happen as a results of electrochemical activities in which the amino acid react with ions of the metals which supported by the Pearson theory where the amino heavy strong affinities to ionic soft base and generally, heavy metals such Lead (Pb^+), Silver (Ag^+), Mercury (Hg^{2+}) etc . These ions are grouped according soft base ions tend react with amino group. The lead is generally classied as the both hard and soft based ion that can easily be react with the donor atoms. As shown table above the water sample contain some degree of lead ions present, this results result was obtained from the atomic absorption spectroscopy (AAS). The The selective of the sensor to the heavy metals ions as a result of membranes, electrochemical interferences creating potentiometric effect across the sensor surface, that will lead to heavy metal detection

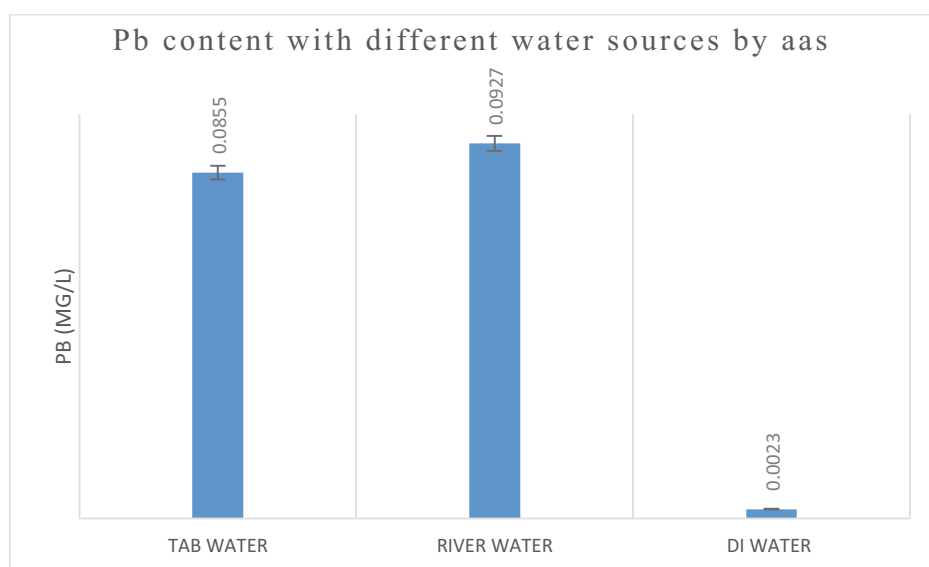


Figure 9. Show comparison of Barred SiNW with 4 different waters collected in 4 different

Generally, the traces amount of metals are common in water, however, it quite harmful, if exceed beyond the standard limits. Especially lead because is a highly toxic metal whose widespread use has caused extensive environmental contamination and health problems, the contamination of lean to environment and ground water highly cmmmon compared to other heavy metals because lead is found every aspect industrial activities, lead used in battery, cosmetics. That why the traces of the lead are always high. For example, the table above is the measurements contacted on three water samples namely Di. Water, tap water and river water, both river water and tab water.

4. Conclusion

The study demonstrated, aptes modified silicon nanowires sensors for electrochemical detection of Pb^+ ions, this does not only enhanced the silicon nanowires but also successfully discriminate the three samples with different electrical behavior. In conclusion, Silicon nanostructure were prepared using cheap

and in house technique developed in INEE. Amino-functionalized Si nanowires were tested against heavy metal lead (Pb). The device was fabricated via dry oxide etching approach with control oxygen flow rate in oxidation furnace, network of uniform Si nanowires were successfully fabricated. The device was functionalized by (3-aminopropyl)triethoxysilane (APTES) to save as a sensor to heavy metal. Due to the silicon electrochemical response toward heavy metal ions, linear response to three different source of water were observed. The results indicated, Pb can be detected with high precision. Furthermore, confirmation were demostarted using atomic absorption spectroscopy instrument (AAS) to determine the level lead content in each water source. Three sources of water were tested: Tab water (H₂O), River (H₂O), DI(H₂O) and 0.0859mg/L, 0.0929mg/L, 0.0023 mg/L with ~ 52pA, 60pA and 70pA current response respectively. Thus, with this high capability to discriminate water samples, the sensor potential to be employed for an effective heavy metals detection and can further be extended for large sensor network in water treatment plant.

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