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To cite this article: Nuri A KH Ehfaed *et al* 2018 *IOP Conf. Ser.: Mater. Sci. Eng.* **454** 012180

IOP Conf. Series: Materials Science and Engineering **454** (2018) 012180 doi:10.1088/1757-899X/454/1/012180

Functionalization of Si Nanowire Surfaces to Create Interactive Mechanism for Heavy Metals Detection Application

Nuri A KH Ehfaed[1], Tijjani Adam[1,] Mohammed Mohammed[2], Omar S Dahham[1] and U. Hashim[3]and Nik Z. Noriman[1], Almahdi R. Rabia[4]

[1]Faculty of Engineering Technology, Kampus Uniciti Alam Sg. Chuchuh, 02100 Padang Besar, Universiti Malaysia Perlis (UniMAP)
[2]Center of Excellence Geopolymer and Green Technology, School of Materials Engineering, Universiti Malaysia Perlis, 01007, P.O. Box 77, D/A Pejabat Pos Besar, Kangar, Perlis, Malaysia
, Perlis, Malaysia
[3]Institute of Nano Electronic Engineering, Universiti Malaysia Perlis, 01000 Kangar, Perlis
[4]School of Environmental Engineering, Universiti Malaysia Perlis, Kompleks Pusat Pengajian Jejawi 3, 02600 Arau, Perlis, Malaysia

Abstract: Silicon (Si) nanostructure was prepared using a cheap and in-house technique. The device was fabricated via dry oxide etching approach with a controlled oxygen flow rate in an oxidation furnace, a network of uniform Si nanowires were successfully fabricated. The device was functionalized by (3-aminopropyl) triethoxysilane (APTES) to serve as a sensor for heavy metal detection. The amino-functionalized Si nanowires were characterized based on variation SPX spectra of four different element that, O, OH, NH_2 and Si-O-NH_2. Three major peaks in the C 1s region one at 289 eV is were detected indicating for the Si-O attaching, values of 287eV 30 for Si-OH, 285eV for NH_2 and 83eV for Si-O-NH_2 representing hydroxide, amine and modified silicon surface that that established nitrogen atoms (-NH2, -NH-). Thus, the study identifies different chemical environments to identify heavy metals in water.

1. Introduction

Electrochemical sensing of heavy metal ions with inorganic, organic and bio-materials are currently occupying the mind every researching working in this field [1-5]. (Cui et al, 2015) have hypothesized in study that electrochemical sensor has short analytical time, low power cost, high sensitivity and easy adaptability for in-situ measurement. When they were summarizing and discussing recent achievements in electrochemical sensing of heavy metal ions within organic and bio-materials using modified electrodes. 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[6-9]. 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. 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) [10-15]. Izuan et al, 2013 claimed that the silicon nanowires having the smallest dimension, SiNWs

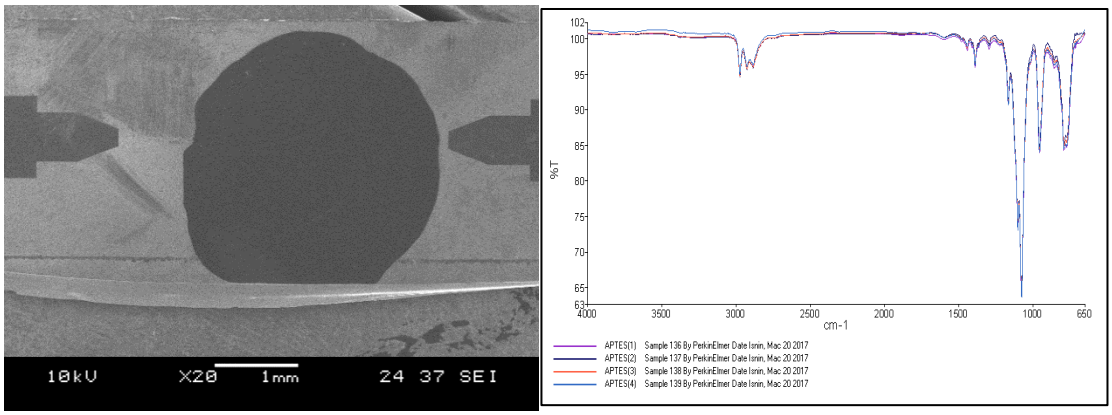
exhibited good electron transfer in detection because the accumulation of charge in SiNWsdirectly occurswithin the bulk of material resulting in fast response of detection. Their study was aim at providing insight on the synthesis of silicon nanowires and provides details information on the potential application of chemical and biological sensors based on SiNWs and they further reveaalled in their study, the posibility of SiNWs surface modification and they its noticed that the surface modification of SiNWs with metal nanoparticles such as gold nanoparticles (AuNPs) and silver nanoparticles (AgNPs) reveal entirely new generation of sensor with novel sensing material electrodes with excellent catalytic activity and high conductivity that can greatly enhance the performance of sensors parameters in terms of sensitivity and selectivity eqaually reliability (Izuan et al, 2013)[16-22]. They claimed that the integration of SiNWs as sensing platform for various analysis with this nanomaterials has great interesting in future for fabrication of miniaturized sensor devices due to their unique properties such as electrical and optical properties (Izuan et al, 2013) .. According to them, the electrochemical and electrical detection showed a great promise in realizing a miniaturized sensor based on SiNWs due to its advantages including high detection, portability, and simplicity of the procedure. However, this advancements come with challenges such viable farbication techniques especially for the bottom up methods because its very hard to develop very precised structures with designed geomerty (Izuan et al, 2013) [23-28]. Hikmat and Billy (2010) have discussed and summarized that biosensors offer label free detection of bimolecular interactions with applications in environmental safety, bioterrorism, biomedical research and drug discovery Hikmat and Billy (2010). Several designs can detect bimolecular interactions and they summed of that biosensors are immensely useful in many different applications and future research aims at improving the sensitivity and throughput of these devices for greater reproducibility and applicability to larger sets of data acquisition Hikmat and Billy (2010). They found out that, the progress in chip design with smaller dimension has allowed for smaller sample volumes, not only to save valuable samples, but also to increase rates of the reactions by reducing diffusion distances. Advances in computer automation and the software to analyze the exquisite data that arise from the discussed methods has also played an important role in greater reproducibility and easier sample handling with all four of the biosensor types capable of employing autosamplers[30-40]. 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[41-45]. 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. Nanoscale silicon has attracted considerable interest due to its potential to impact broad areas ranging from electronics, photonics and renewable energy to biomedical sensing. Significant efforts have been devoted to develop new silicon nanostructures, including quantum dots, nanowires or porous silicon. Silicon nanowires, in particular, have been in the focus of intensive research over the past decade due to their unique one-dimensional physical morphology and the associated electrical, mechanical and therm flexible large area electronics, thermoelectric, photovoltaics, battery electrode, and electronic biosensing [46]. However, with an indirect band gap, silicon nanowires can hardly be explored as an optically active material for functional optoelectronics. On the other hand, silicon quantum dots and porous silicon are well-known for their ability to exhibit luminescence in the visible range due to strong quantum confinement effects, and have attracted significant attention for silicon based optoelectronics including light-emitting diodes and lasers. A combination of these two features and the formation of both electrically and optically[47-49]. 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,

2. Methods

Surface modification of the si-nanowire surface brings the new chemistry for the sensor which make suitable platform for various chemical and biospecies detection. The process involved two major steps, the surface treatment (modification) and, immobilization (probe attachment). The methods start with preparing the nanowires surface with using DI water and acetone. This was dried on hot plate for 2 min. The prepared surface was ready for the treatment, prior to the surface treatment the APTES was diluted in 5% 3-aminopropyltriethoxysilane (APTES g/g) in a mixture of 95% ethanol and 5% water. The diluted solution gradually dropped on the prepared si-nanowires and dried for 3 hrs in room temperature. After this, it was washed with IPA mix with ID water to remove any unwanted impurities and unreacted APTES, the APTES-functionalized nanowires surface was characterized with SPX to study composition.

3. Results and discussion



(a) (b)

Figure 1. (a) APTES modified Silicon nanowire (b) Figure 4.24: FTIR spectra of APTES modified silicon

As can be seen in this figure 1a, it obvious that APTES is present on the silicon nanowires between the two electrodes. FTIR analysis as shown in figure 1b, was carried out to investigate any chemical composition of silicon. It can be observed that no extra peak appeared to show any chemical difference among all the functional group silicon and a new functional group will appear when any chemical changes occur during surface modification.

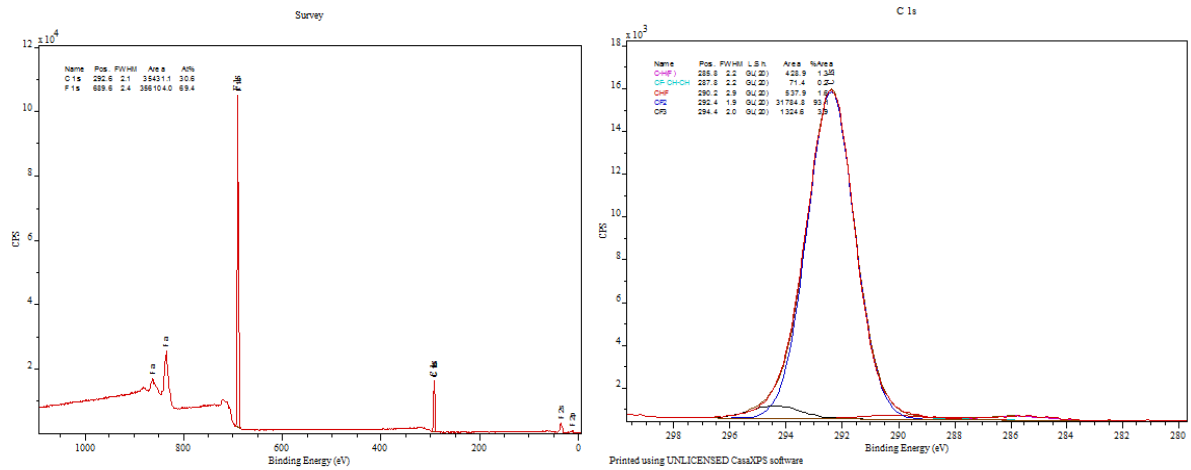


Figure 2. Show (a) the mechanism of oxidation for silicon with amine (b) Figure 26: XPS Si 2p region of a Si sample with a NH_2, comparing a fresh sample and the same sample after one month no differences are observed

The reactions in solution phase involve creation of a dangling bond after Si-OH bond cleavage and electrophilic attack of doubly bonded hydroxyl bond Si-OH bond during drying exposure and in solution phase getting dry , the reaction might proceed by the dissociation of a hydrogen atom. The silicon surface which probably interacts with an amine molecule resulting in a Si-N bond after another hydrogen atom cleaved from the amine group (-NH2), or it might involve only the dissociation of the N-H bond. The electronegativity of the –NH group, in case of the Si-N bond, is high and renders the silicon atom positively charged, however, cannot be attacked directly by aqueous oxygen due to steric hindrance but renders oxidation at the subsurface silicon atom favorable due to an inductive effect . This attack is more favorable in the case of a dative bond (Si-+NH2) and leads to a weakening of the Si-Si bond, while increasing its length and makes the oxygen atom to bridge between two silicon atoms possible figure 2a. The silicon surface can be prepared by using APTES without protecting the tail group (NH_2) under optimal reaction conditions. compares the Si 2p regions of freshly modified silicon surfaces by OH and NH_2. The small amount of surface oxidation can be seen in the case of the on the surface that might be due to the interaction of the NH_2 group with the silicon surface inducing small subsurface oxidation.

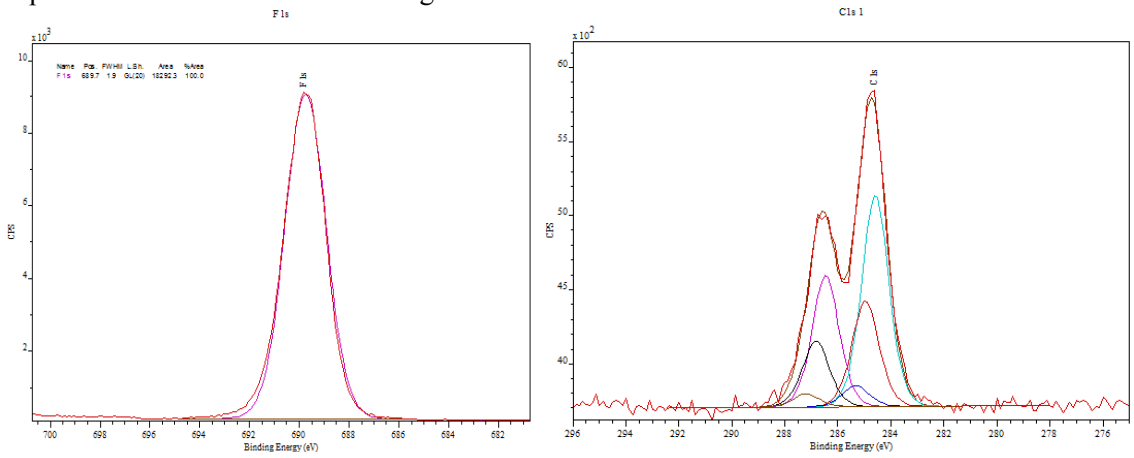


Figure 3. (a) correlating the high resolution XPS spectra of Si-OH bonded and Si-O (b) Show the SPX spectra comparison NH2 and OH, the NH_2

The figure 3 correlating the high resolution XPS spectra of Si-OH bonded and Si-O It can be concluded that the oxidation peak in survey spectrum for NH2 is high due which adsorbed water and oxygen congaing impurities attached to free amine terminated surface and also extensive sub-surface oxidation caused by amine groups attached to silicon in the presence of trace aqueous oxygen where the back-bond of silicon is only oxidized and not the surface silicon atoms leaving Si-H bond or Si-O bonds intact and any functionality on the silicon will remain available for immobilization. Figure 3 show the SPX spectra NH2 and OH, the NH_2 surface has more ions contents as shown by the SPS spectra, this is probably because of the fact that the amine terminated surfaces are more susceptible towards adventitious impurities from the environment as well as over exposure to radiation during drying might brough some oother impurities bond in the amine groups of the resulting in Si-O-OH attachment. The figure show the spectra of three element that, O, OH, NH_2 and Si-O-NH_2 . Three major peaks in the C 1s region one at 289 eV is most probably due to the Si-O attached, the second at 287eV 30 due to Si-OH, 285eV due to NH_2 and 83eV is due to Si-O-NH_2, peaks representing amine that it can be assumed are attached to nitrogen atoms (-NH2, -NH-), which have two different chemical environments, the intensity of the peak at 288 eV might be attenuated due to adventitious impurities.

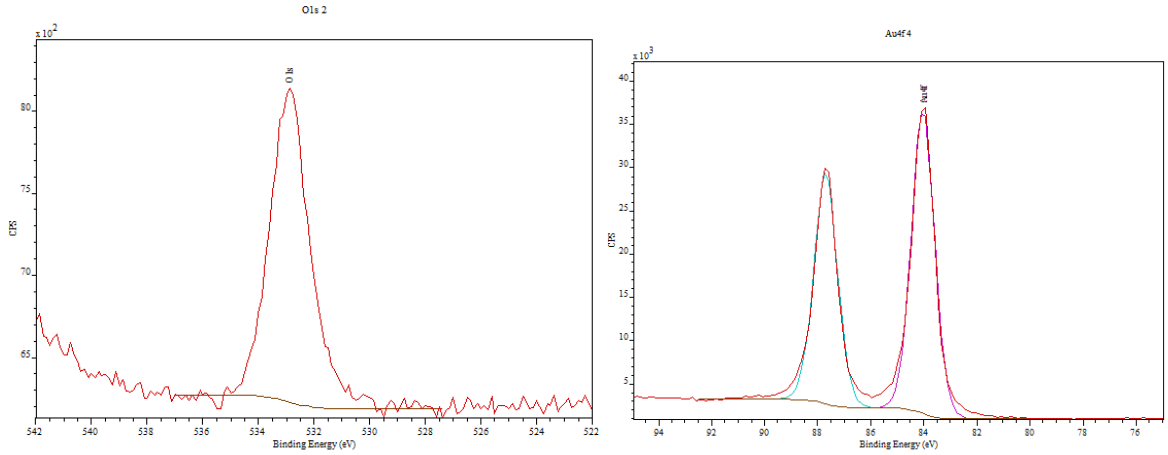


Figure 4. (a) Silicon surfce total reaction of the probe amine (b) shows spectra of as-prepared silicon nanowire surface consisting of conventional Si-OH bonded layers and a Si-NH_2 bonded layer

The figure 4a show the total reaction of the probe amine and it can see only a very small region of the oxygen peak at 534 eV might be due to the fully developed surface silicon dioxide where as a fit at 532 eV is probably due to a small amount of sub-surface oxidation,26 but mostly from the top surface impurities getting adsorbed onto the surface during wash process. The O 1s XPS signal could arise from many sources, including adsorbed solvent from the wet chemical preparation techniques. Figure 4b : shows spectra of as-prepared silicon nanowire surface consisting of conventional Si-OH bonded layers and a Si-NH_2 bonded layer. The present of the Si-OH might be due to two reasons, these include, the nucleophilic nature of amine groups which might act as a catalyst to oxidize the silicon surface utilizing traces of oxygen in solution and the hydrogen bonded water on the surfaces increase the oxygen content in XPS spectrum.

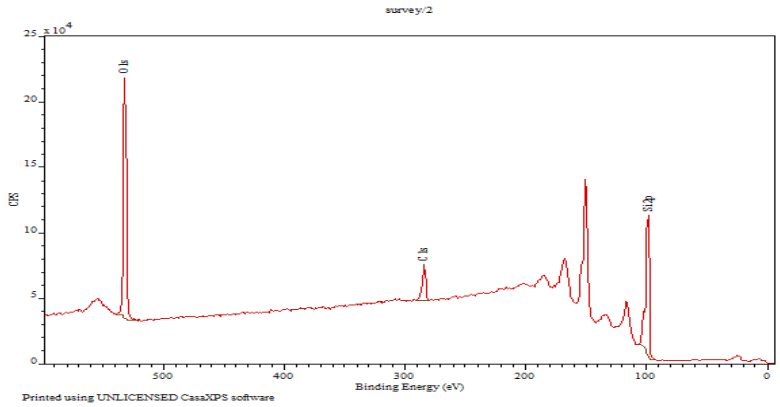


Figure 5. XPS spectrum of APTES modified on silicon nanowire surface

The figure 5 show SPX spectrum of the APTES on planar Si nanowires, small peaks at 550eV, 270eV, 150eV and 110Ev shows relatively small amounts of oxide which is approximately equal to that for 50Å shown in filmetrics data. Region of the XPS spectrum for the amine bonded monolayer where the oxide peak at 270eV is nearly absent, however, in contrast, the O 1s region of the same spectrum shows sufficiently prominent oxygen peak and amine bond.

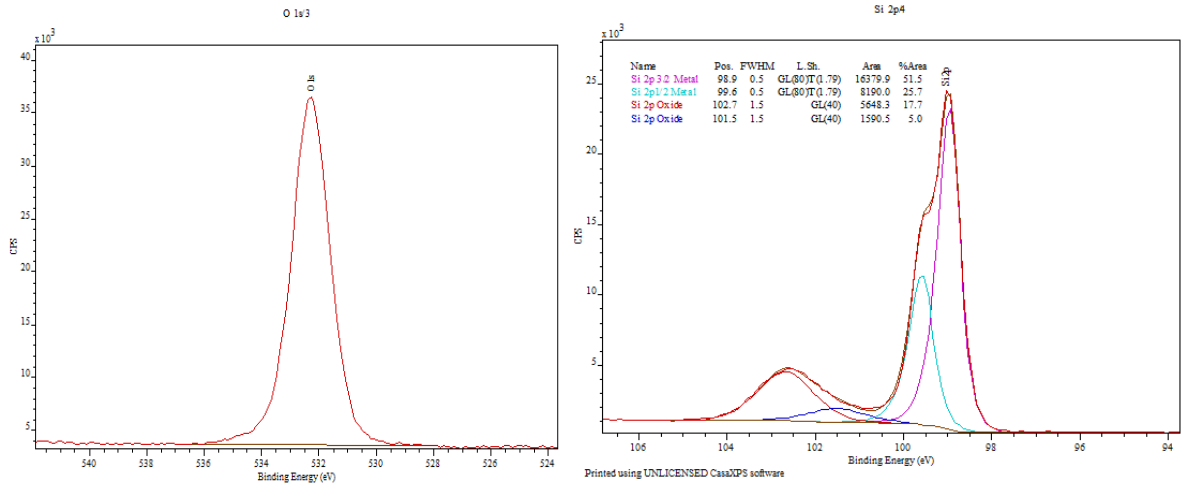


Figure 6. Show (a) the reaction of APTES with hydroxide (OH) terminated silicon in solution phase (b) High resolution XPS spectrum of the amine on silicon

The reaction of APTES with hydroxide (OH) terminated silicon in solution phase under it is assumed hydrogen atom dissociates from the amine group creating a free dissociated amine radical that reacts with the hydrogen terminated silicon surface. According to their proposed reaction mechanism, a surface is not necessarily exposed directly for amine adsorption avoiding degradation of the probe. Figure 6 show Si 2p region of the spectrum continuum fitted peaks at 99.9 eV, 100 eV, 101.8 eV and 103 eV. The silicon oxidation state for the Si-N bond should be at 101.78 eV,33 however, it is difficult to distinguish in all samples from the Si-H and Si-O regions. The Si 2p region starting from 101 eV to 104 eV can be fitted with two major peaks one at 102.76 eV due, to (Si—N) and the second at 103.5 eV representing the (Si-O) bond which is fully developed SiO2 and a third fit at 104 eV is most probably due to a Si-N dative bond (Si-+NH2) due to higher energy requirements to remove an electron. However, the Si-N remains intact, N 1s is compared with N and a monolayer of adsorbed amine with free amine groups remain available for further conjugation of biomolecules on the sub-oxide (Si) surface..

4. Conclusion

To react with heavy metal ion with SiNW-based devices, APTES has been the most reliable silane-based coupling agent. However, enabling immobilization of APTES to chemically bind amine-terminated biomolecules comes with some difficulties because silane compounds often react with other foreign elements via cross-linking of the alkoxy units, resulting in disturbing the sensitivity of sensor devices. Thus, this study, able to present the result of APTES silicon nanowire, the SPX results show a very clear and pure APTES immobilized probe that could be reactive to the heavy metals ions.

5. Acknowledgement

The author would like to acknowledge support from the Short-Term Grant under a grant number of 9001-00526, Universiti Malaysia Perlis (UniMAP).

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