Current-Voltage Characteristics of Nb$_2$O$_5$ nanoporous via light illumination

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Abstract. This work discussed the effect of light on I-V characteristics of anodized niobium pentoxide (Nb$_2$O$_5$) which formed nanoporous structure film. The structure was synthesized by anodizing niobium foils in glycerol based solution with 10 wt% supplied by two different voltages, 5V and 10V. The anodized foils that contained Nb$_2$O$_5$ film were then annealed to obtain an orthorhombic phase for 30 minutes at 450°C. The metal contact used for I-V testing was platinum (Pt) and it was deposited using thermal evaporator at 30nm thickness. I-V tests were conducted under different condition; dark and illumination to study the effect of light on I-V characteristics of anodized nanoporous Nb$_2$O$_5$. Higher anodization voltage and longer anodization time resulted in higher pore dispersion and larger pore size causing the current to increase. The increase of conductivity in I-V behaviour of Nb$_2$O$_5$ device is also affected by the illumination test as higher light intensity caused space charge region width to increase, thus making it easier for electron transfer between energy band gap.

1. Introduction

Current technology is in need of a new material breakthrough to catch up into the ever-vast trend of modern devices. One of the potential material that has yet to be fully discovered is Niobium (Nb). It has started to grow in interest with recent years due to its relatively abundant in nature, and interesting structural phases [1]. The oxide form of niobium, Niobium Pentoxide (Nb$_2$O$_5$) had been researched as early as 1940s because of its many polymorphic forms and from there the exploration of Nb$_2$O$_5$ kickstarted. Its characteristics of having high corrosion resistance, thermodynamically stable with unique performance of the energy band diagram and the identification of crystal phases had made the material to be further investigated [2].

Some of the earliest applications of Nb$_2$O$_5$ as functional material were the in the production of sensors, acting as catalyst and used in electrochromism [3]. The first level of Nb$_2$O$_5$ research was in either in thin layer, bulk or suspensions but recently the synthesis process of producing various Nb$_2$O$_5$ morphology had grown in popularity. Since the advancement of nanotechnology, Nb$_2$O$_5$ research had been focused to a more complex study of morphology.

Several synthesis methods have been reported to obtained nano-structured Nb$_2$O$_5$, in which some of them are anodization [4,5], sol–gel dip-coatings , hydro-thermal, pulsed laser deposition and electrodeposition [2,3,6–8]. Due to anodization's capability of forming highly porous and ordered oxide morphology to Nb$_2$O$_5$, it is one of the most widespread nano-fabrication methods. Anodization
method of Nb₂O₅ synthesis is in the most peak interest among others are also due to its advantages of having low fabrication cost and controllable film thickness.

The growth and morphology of the anodic oxide film strongly depends on the applied anodization potential, the composition of the electrolyte, the electrolyte temperature, and the anodization duration. The as-anodized Nb₂O₅ films are mainly amorphous and require a post-annealing treatment to be converted into highly crystalline and stoichiometric Nb₂O₅ [9,10].

For Nb₂O₅ to be applied as devices, there are various ways of fabrication techniques with different atmospheric exposures can be done. Different medium used in the fabrication gives a huge role in contributing to its performance, particularly current-voltage (I-V) characteristics in electronic devices. I-V behaviours can be affected by atmospheric exposures such as temperatures, external currents, radio frequency and also the illumination of light [11–13]. The light presents an effect on I-V behaviour of the device by changing the width of the space charge region (SCR). This change directly affects the reverse bias I-V curves by increasing the current values and the slope of the curves. The photodetection performance of the devices tends to increase with the increasing light power due to the increasing SCR width [14].

To the extent of author’s knowledge, currently there is no research or publication towards Nb₂O₅ I-V characterization to the effect of light illumination. In this paper, the project focused on the I-V behaviour of anodize Nb₂O₅ with different anodization variables on the effect of light illumination. The I-V results showed that there is slight increase of current when samples were being exposed to light.

2. Experimental Procedure

2.1. Fabrication of nanoporous Nb₂O₅

Niobium foils with dimensions of 2.5 cm × 1 cm (99.9% purity, Sigma Aldrich) were gone through ultrasonic cleaning with methanol and deionized water before they were anodized at different time ranging from 2min, 4 min and 6min in different voltage range (5V, 10V and 15V) to produce Nb₂O₅ nanoporous structures. The anodization was conducted using the electrolyte containing 10 wt.% K₂HPO₄ (98% purity, Sigma Aldrich) in anhydrous glycerol (99% purity, Sigma Aldrich), at 180°C as suggested previously from different works. The as-anodized samples were annealed in air at 450°C for 30 min (ramp up/down of 2°C per min). The Nb₂O₅ has many polymorphic forms which gives rise to an interesting series of structural phases.

2.2. Characterization

For structural characterization, Field Emission Scanning Electron Microscopy (FESEM) was conducted to study the surface morphology of the sample using JEOL JSM-7600F. Electrical connections to the Schottky gas sensors were achieved by physically connecting the probes to the deposited Pt metal (top electrode) and Nb foil (bottom electrode). The current–voltage (I-V) measurements were carried out using the two-point probe of Keithley 4200 as they were tested in a dark condition and illuminated from -4V to 4V applied voltage.

3. Results

3.1. FESEM images

The FESEM images in Figure 1 show the surface morphology of the synthesized annealed Nb₂O₅ layer. Looking along the 2-min anodization of Figure 1(a-i), 1(b-i), and 1(c-i), as voltage applied increases from 5V, 10V and 15V, the dispersion of pores appeared to be higher, thus lowering the pore density. The same case is also shown at 6-min anodization of Figure 1(a-ii), 1(b-ii), 1(c-ii). Additionally, if the diameter of pore size is being compared, the diameter size increased as the anodization time increased. At 5V anodization of 2 min, the pore size is at average around 7nm while
the at 6 min anodization, the average pore size is at 9nm range.

For 10V anodization of 2 min, the average pore size of nanoporous Nb$_2$O$_5$ is around 8nm, while at 6 min the average pore size is larger, which is approximately 13nm, an indication showing higher anodization voltage formed higher pore size. The same case also applied on 15V anodization where at 2-min synthesis, the average pore size is around 10nm while at 6-min synthesis the average pore size is 20nm. Figure 1(d) showed the cross-section image of 6 min anodized Nb$_2$O$_5$ at 15V where ‘vein-like’ nanoporous structure can be seen.

Figure 1. Surface morphology in 5V, 10V, and 15V in (a), (b), and (c) respectively with (i) in 2-min and (ii) in 6-min anodization time, and (d) cross-section image of anodized Nb$_2$O$_5$. 
3.2. I-V Test

Figure 2. I-V test results comparison of (a) 5V, (b) 10V, and (c) 15V Nb$_2$O$_5$ anodization of dark VS illumination test, while (d) focuses on overall I-V illumination test result comparison of 5V, 10V, and 10V Nb$_2$O$_5$ anodization.

Figure 2 illustrates a rather similar I-V pattern occurred for Figure 2(a), 2(b) and 2(c). The plots of I-V show preferably linear relation in both dark and light condition, and specifically obtained higher current when the samples were tested under illumination. According to Palatnikov et al [15], light exposure leads to the formation of fractal micro and nanostructures in Nb$_2$O$_5$, which have a significant effect on its thermal expansion, thus making it prone to harbor higher energy that caused it to produce higher current.

Other researchers [16–19] also suggested the same case where photon energy from the light makes the band gap of the material decreases, thus making it easier for the charge carrier to crosses over and increase the rate of current density. The increasing light power increases SCR width of the material thus the photodetection performance of devices tends to increase. These factors can contribute to the conductivity of the film since the flow of electrons is moving faster at higher dispersion of pores and bigger pore sizes [19] since it provides continuous and directional paths for electron transfer. When photon energy is present, electrons gain higher energy to jump across the band gap.

The directional paths transfer also affected the I-V characteristic of the device with different thickness. This can be seen when the voltage synthesis of anodized Nb$_2$O$_5$ is being compared (Figure 2(d)) in which the result suggested lower anodization voltage produced higher current than that of higher anodization voltage.
CONCLUSION

This study presented the current-voltage characteristics of Nb$_2$O$_5$ nanoporous via light illumination. Higher anodization voltage produces bigger pore sizes and larger pore dispersion. Along with longer anodization time, the pore size also increases significantly which leads to encouragement of electron transfer, which in relation produce higher current. The case also follows when I-V is tested on different photon exposure. Compared with those in dark condition, the magnitude of the current is larger under illumination. However, when the current level is being compared between different anodization voltage, lower anodization time produces higher current since it produces thinner layers of Nb$_2$O$_5$. The increasing change in light intensity caused the photodetection performance of the device to increase due to the increasing SCR width, thus producing higher jump rate of electrons to move across the band gap.

ACKNOWLEDGEMENT

The authors would like to acknowledge with gratitude the NANO Electronic Centre of Faculty of Electrical Engineering UiTM for the facilities. This work is supported by Ministry of Education Malaysia (MOE) under the Research Assistant Grant Scheme (RAGS) (Project code: 600-RMI/RAGS 5/3 (6/2015)).

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