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Electrical characterization of a single TiO_2 nanotube by using modified FIB/SEM

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TiO_2 nanotubes have unique properties that originate from their geometric shapes and quantum size effects. Their applications in nanoelectronics, nano-sensors and nano-bio engineering are of great interest in the industry. However, there are still many challenges in terms of nanotube manipulation and electrical characterization. Conventional nanofabrication techniques such as electron beam lithography and physical vapor deposition are not chosen here because the results obtained are often not reproducible and the methods are labor intensive and require multifarious processing steps. The dual beam focused ion beam (FIB), with its unique capability of selected-area deposition, is a great tool for nanofabrication and electrical characterization on nano-size structures and nanotubes [1]. It allows for the formation of complex fabrication with minimal steps and provides high spatial resolution. In this paper, we present results of the *in-situ* and *ex-situ* electrical characterization of single TiO_2 nanotubes by using electrical test structures fabricated by Focused Ion Beam Induced Deposition (FIBID) .

For this experiment, a dual beam FIB (FEI NanoLab200) equipped with a gas injection system (GIS) was used for nanofabrication. In order to measure the electrical properties of the TiO_2 nanotubes, electrical test device structures, fabricated by FIBID, were used. Platinum interconnects were then deposited between the Au/Cr electrodes and the electrical TiO_2 test device structures. TiO_2 nanotubes were pre-formed inside a soluble nanotemplate using atomic layer deposition (ALD) [2]. The prepared solution and the nanotubes were dispersed on top of the SiO_2/Si substrate. Electrodes were patterned on insulating SiO_2/Si substrates. The geometry of an electrode is 50 µm x 50 µm.

To conduct electrical studies, electrical test structures for in-situ characterization were fabricated by FIBID and measured in-situ as shown in Fig. 1. The TiO_2 nanotubes have a diameter of 200 nm, 20 nm thick walls, and are 5 µm in length. Electrical I-V measurement results show semiconducting resistivity in the order of 1 Ωcm, which is much lower than that of stoichiometric TiO_2 bulk (~10^8 Ωcm) [3]. When the current level reached the saturation point, nanotube degradation was observed, which confirms that a current flowed through the nanotube and platinum and gallium contaminations were avoided. It is known that TiO_2 is highly sensitive to its surrounding environment and its conductivity can be affected by oxygen vacancy. As illustrated in Fig. 2, the conductivity of single TiO_2 nanotube measured *in-situ* is higher than that in atmosphere, which means there are more vacancies in *in-situ* because TiO_2 nanotubes are subjected to a lower oxygen pressure. Additionally, electron beam exposure can be considered as an important factor which affects the properties of the TiO_2 nanotube. When the TiO_2 nanotube was exposed to an electron beam, the conductivity of the TiO_2 nanotube increased as shown in Fig 3. However, the conductivity did not return to its original level, even though it was discharged for a week. This indicates that electrochemical reactions such as oxygen reduction occurred when the TiO_2 nanotube was exposed to the E-beam, which leads to the increase of oxygen vacancies. Essentially, the electrical characterization of a single TiO_2 nanotube by using FIBID fabricated electrical test structures has been successfully demonstrated. The semiconducting property of TiO_2 nanotubes has been discovered and their reaction to the atmosphere and electron-beam used in FIB are observed.

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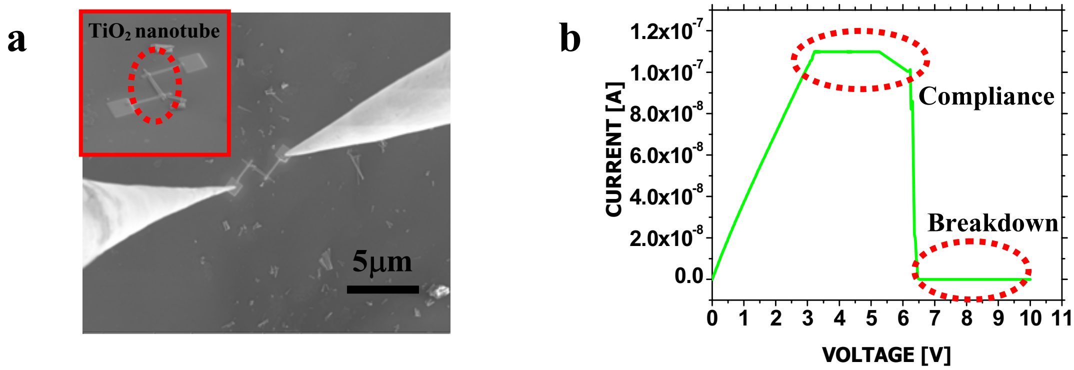

FIG. 1. (a) SEM image of W probes and TiO_2 nanotube electrical test structures fabricated by FIBID. (b) TiO2 nanotubes with 200 nm diameter and 15 nm thick walls show semiconducting resistivity of an order of 1 ·Ωcm.

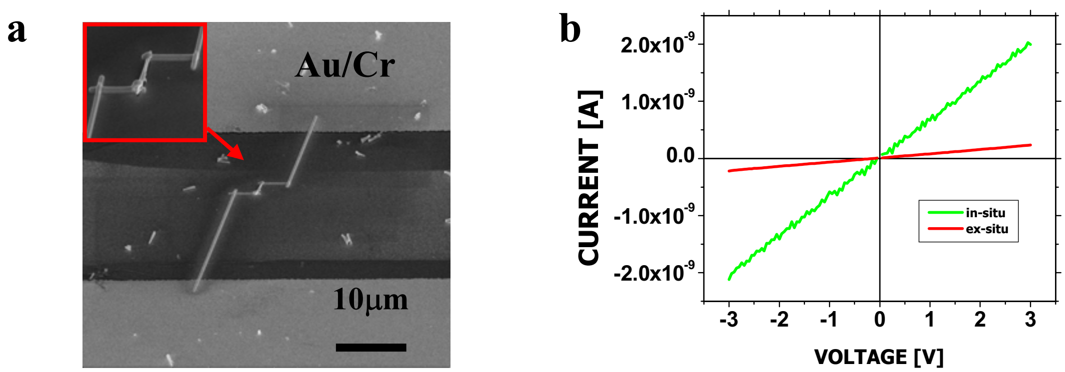

FIG. 2. (a) SEM image of TiO_2 nanotube test structure with the Au/Cr electrodes. (b) I-V measurement results: *in-situ* vs. atmosphere.

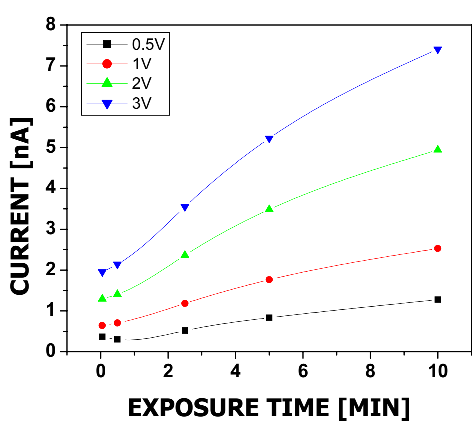

FIG. 3. I-V behavior with respect to E-beam exposure time.

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