The development of reproducible in situ electrical biasing of semiconductor materials using piezo-controlled electrical contacts and chip based systems.

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In this presentation we will show how different approaches have been developed for performing in-situ electrical biasing using different types of specimen holders. Piezo-based specimen holders, such as the Nanofactory and our recently acquired Hummingbird version use a movable contact that can be placed onto a desired region of interest with nm-scale resolution. Alternatively, chip based systems can be used where a device is electrically connected by using metal deposition in the focused ion beam tool such as the Protochips Aduro system. A working methodology has been developed for each type of specimen holder which will be discussed as well as the advantages and disadvantages of using each type of method.

To illustrate that these methods can be applied to real specimens, we will focus on two examples. Firstly a simple p-n junction that is studied by electron holography to measure the increase in potential across the junction as the specimen is reverse biased. We will also present results from a Pt/SrTiO3/Nb:SrTiO3 system is which is considered to be a model for the study of resistive memories [1]. This device can be switched electrically from a low resistive state (LRS) to a high resistive state (HRS) through electrical control. We will present aberration-corrected scanning (S)TEM and electron energy-loss spectroscopy (EELS) measurements of the composition and bonding that are obtained during cycling. It is thought that changes in the oxygen concentration underneath the top electrode are responsible for the switching and as such it is important to be able to visualize the movement of oxygen vacancies as the devices are reversibly switched.

Figure 1(a) shows a STEM image of device comprising a Nb:doped SrTiO3 substrate (bottom electrode), a 18-nm-thick Sr-enriched SrTiO3 (Sr:STO) film and a 10-nm-thick Pt (top electrode). The substrate of the device has been electrically connected and the movable probe is placed onto a Au top contact far from the region of interest. Figure 1(b) shows that electrically area switching is expected as the device is cycled between states and (c) shows a STEM image of the device after switching where the top 4 nm of the has been altered. The EELS results shown in (d) suggest that there is a change in the Ti edges as the specimen switches into a LRS under the top contact which is reflected in the Ti-O maps in (e) and (f) which show the device in a LRS and HRS respectively confirming the presence of area switching. Figure 2(a) shows a similar specimen being attached to a chip type device and (b) shows a STEM image revealing many grain boundaries, therefore this device should preferentially exhibit filamentary switching. This is observed as the device is cycled in-situ in (c) where three decades of resistance switching are observed. Figures 2(d) and (e) show oxygen maps of the region of interest as the device is cycled between the different states. We will discuss issues such as specimen heating and strain and how the different methodologies can affect the experimental results.

[1] R Waser and M. Aono, Nature Materials 6 (2007) p. 833.

[2] D. Cooper et al, Advanced Materials 29 (2017) 1700212

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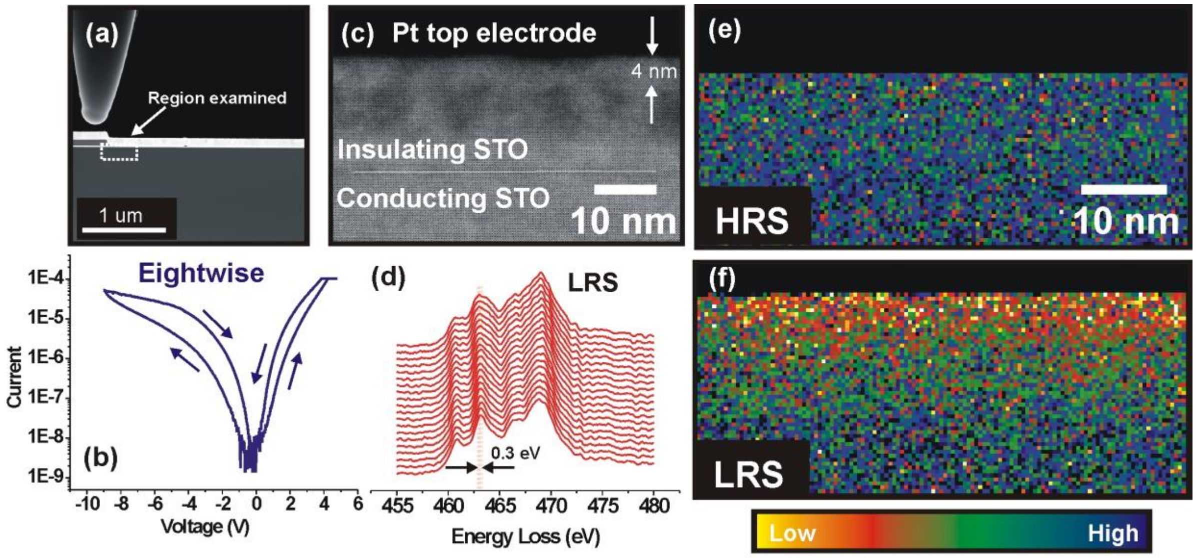

Figure 1.(a) A Pt/STO/Nb:STO stack is connected using a Nanofactory piezo holder (b) which is cycled in the TEM indicating area (eightwise) switching. (c) STEM images show that there is redistribution of material in the top 4 nm of the STO region. (d) Ti spectra show changes in the edges which can be linked to the Ti-O bonds or oxygen vacancies. (e) and (f) show Ti-O bond maps for the HRS and LRS state showing that there is a concentration of oxygen vacancies under the Pt top electrode when the device [2].

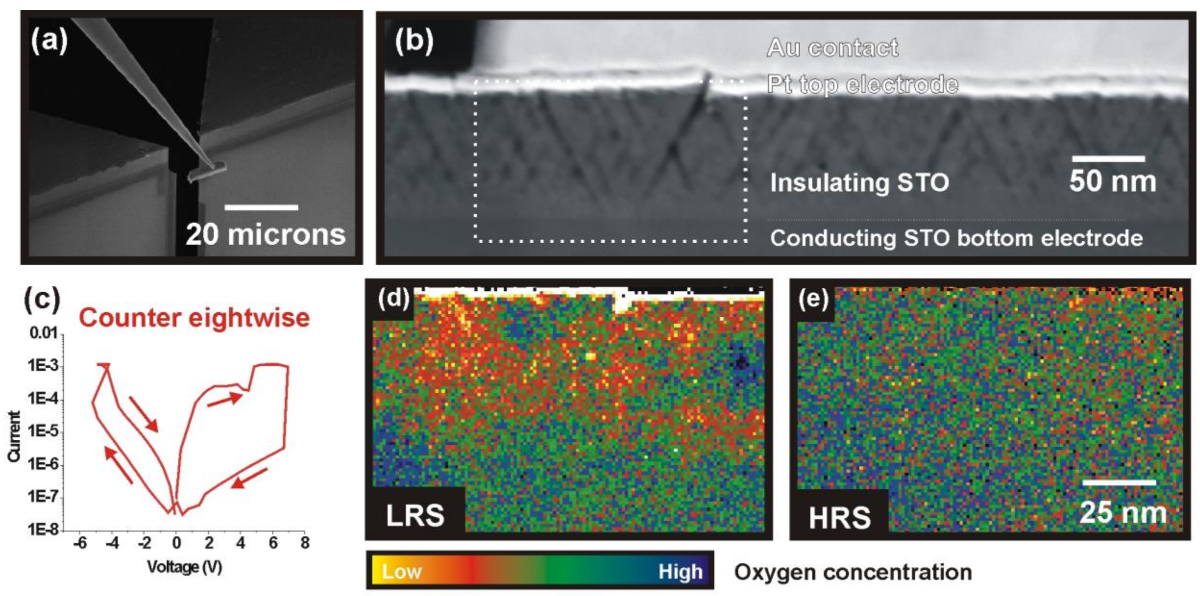

Figure 2.(a) A Pt/STO/Nb:STO stack is connected to a chip in a Protochips Aduro 500 holder. (b) STEM images show the structure of the specimen which (c) is cycled in the TEM and this time filamentary (counter eightwise) switching is observed, (d) and (e) show oxygen maps for the LRS and HRS states.