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STEM Video of Electronically-Driven Metal-Insulator Transitions in Nanoscale NbO_2 Devices

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Above a characteristic temperature, some insulating materials, typically metal oxides, undergo a reversible transition into a low resistivity phase. Such metal-insulator transition (MIT) materials are being studied for possible applications in electronic devices, as this transition can be triggered locally via Joule-heating. Among the known MIT materials, niobium dioxide (NbO_2) is of particular interest because it has a transition temperature (1081 K) that is above typical operating temperatures for semiconductor electronics applications.

In bulk NbO_2 the MIT occurs due to a crystallographic transformation between rutile and distorted rutile structure above and below the transition temperature, respectively [1]. In an NbO_2 thin film device under voltage bias, this transition produces a volatile and reversible low resistance state above a threshold voltage. Because of this property, NbO_2 has been suggested as a possible selector material in crossbar arrays of resistive memory elements as a method for preventing "sneak current" switching. Though several bulk heating and biasing studies have been performed, the MIT in nanoscale devices under local Joule heating is not well understood. Here we report on imaging the MIT *in situ* in nanoscale, horizontally-aligned Pt/NbO_2/Pt devices with multi-detector scanning transmission electron microscope (STEM) imaging.

Our horizontal devices (Figure 1, left inset) allow TEM imaging of an MIT material between biasing electrodes. Ti/Pt (5/25 nm) electrodes (50 nm wide with 30 nm between electrodes) were fabricated on an electron transparent Si_3N_4 membrane using electron beam lithography. 50 nm of NbO_2 was deposited over the electrodes via pulsed laser deposition at 700^{o}C substrate temperature in 2 mTorr Ar+O_2 growth atmosphere with 1% O_2 content using an NbO_2 ceramic target (AJA International.). Film quality and thickness were assessed with x-ray photoelectron spectroscopy, x-ray diffraction, and x-ray reflectometry, confirming the major NbO_2 phase, which forms with an additional top Nb_2O_5 layer of ~2 nm due to air exposure [2]. STEM video was obtained simultaneously from 3 different detectors, collecting electrons at low (bright field or BF), intermediate (dark field or DF), and higher scattering angles (high-angle dark field or HDF). Each detector is sensitive to different contrast mechanisms; diffraction contrast is more apparent at low and intermediate scattering angles while thickness and atomic number *Z* contrast dominate at higher angles.

Current-voltage (IV) data acquired *in situ* show an electroforming step and two subsequent transition cycles (Figure 1, left). The bias voltage was ramped slowly (~50 mV/s) and the current was limited to 40 μA. After an initial electroforming step, the device exhibited reversible, polarity-independent threshold switching at ~1V. Such switching repeated over multiple cycles. We observed similar behavior in several devices biased *in situ*. Despite our non-standard geometry, the devices switched with an electric field of ~3x10^7 V/m and ~1 nW/nm^3 power density, values comparable to those of vertical

Pt/NbO_2/Pt devices [4]. The hysteresis in the IV curves is evidence of the expected MIT switching behavior.

The structure of the Pt/NbO_2/Pt device changed noticeably during the forming step (Figure 1, right). In each detector the electrode geometry appears unchanged by the transition, ruling out the formation of a conducting channel through mass transport of electrode material [3]. In the BF and DF channels, significant NbO_2 grain reconfiguration can be seen between the electrodes. In the BF image a ~50 nm wide dark grain appears overlapping with the lower contact, and is presumably situated in a plane above the electrodes. The DF channel shows that, while regions of NbO_2 far from the electrodes change very little during forming, several grains appear in the region running along the length of, and ~20 nm to the left of, the electrodes (encircled in Figure 1, right). This reconfiguration suggests that electroforming occurs in a region surrounding the electrodes, and not just between them, thus the forming transition is likely triggered thermally, rather than via field-driven processes. Subsequent MITs drive more subtle reconfigurations (STEM images not shown) of the NbO_2 grains in the regions affected by forming.

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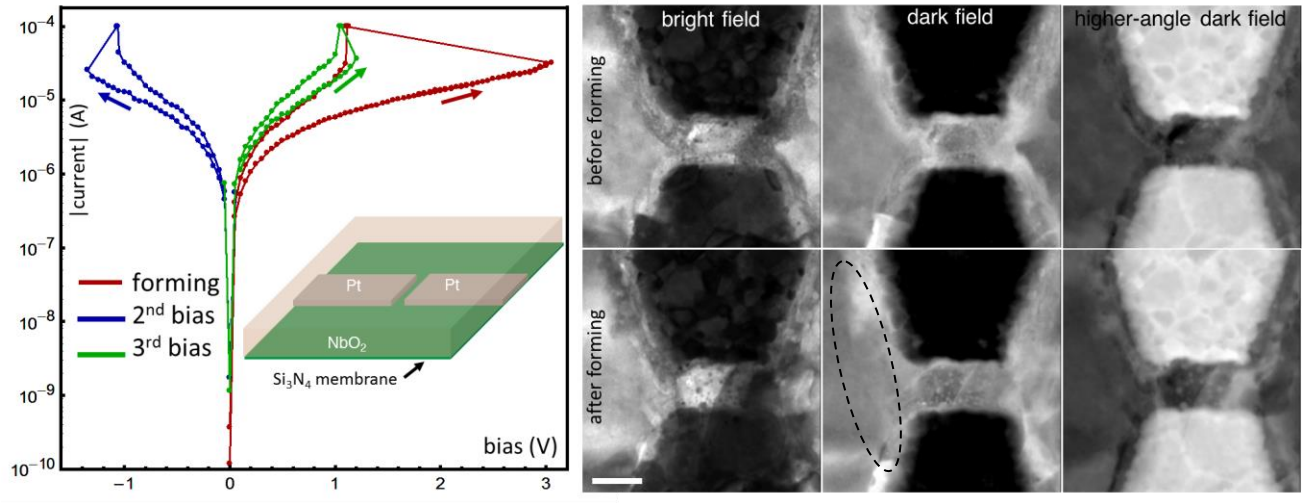

Figure 1. (left) Current vs bias voltage plot for a forming and two subsequent transition cycles for a device biased *in situ*. The colored arrows indicate the point ordering in each cycle. Inset: device schematic. (right) STEM images from 3 different detectors showing the device before (upper row) and after (lower row) forming. The dashed oval encircles features outside the expected switching region that changed significantly during electroforming. Scale bar is 25 nm.