Automated in situ transmission electron microscopy experiments

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In situ transmission electron microscopy (TEM) involves the application of a stimulus to a specimen in the TEM while changes to the specimen are recorded using imaging, diffraction or spectroscopic techniques. However, in most previous *in situ* TEM studies the apparatus that was used to apply a stimulus did not communicate with the software or hardware that was used to control the TEM and collect data.

Important criteria for *in situ* TEM experiments include minimisation of irradiation dose and avoidance of user bias, resulting in the need to work quickly - and ideally in an automated way. A direct interface between a setup used to apply a stimulus and an interface used to control the TEM is therefore crucial. We have implemented plug-ins for Digital Microcrograph (DM), which can be used to communicate directly with a GPIB bus compatible setup (Fig. 1 a) and external Labview-based software that can then be used to control the stimulus applied to the specimen (e.g., temperature regulation). Values of the applied stimulus and signals measured from the specimen are recorded and added to the tags and titles of TEM images.

We have studied silicon oxide-based resistive switching devices *in situ* in the TEM using a movable W needle and recorded bright-field (BF) TEM images with different voltages applied to the specimen (Figs 1 b-d). A DM script was used to apply a voltage ramp and to measure the current flowing through the sample in an automated way for each applied voltage. By using this approach, we were able to follow the formation and destruction of a conductive path across the SiO_x layer and to correlate it with a measured change in conductivity.

A second experiment involved *in situ* electrical biasing of a solar cell and recording a map of electron beam induced current (EBIC) inside the TEM. DM plug-ins were used to record the current generated by the electron beam while scanning the active layer of a µc-Si:H solar cell (Fig. 2 a). The same script was used to measure the current across the sample as the electron beam was scanned across the specimen and a voltage applied to the solar cell. Simultaneously acquired scanning TEM and EBIC maps are shown in Figs 2 (c-d).

A further *in situ* TEM experiment performed on a biased solar cell involved the acquisition of off-axis electron holograms to determine changes in electrostatic potential across the active layer. An external stimulus such as an applied bias can be applied to such as specimen to remove the unwanted mean inner potential contribution from the results. For each applied voltage, a hologram was acquired from the area of interest on the specimen, the stage was moved to record a vacuum reference hologram and it was then returned to the same sample area. This approach was used to record a series of amplitude and phase images from electron holograms of an electrically biased Si:H solar cell (Fig. 2 e) and to extract phase profiles across the top ZnO contact, the p-doped Si layer and the amorphous intrinsic layer (top right of Fig. 2 e).

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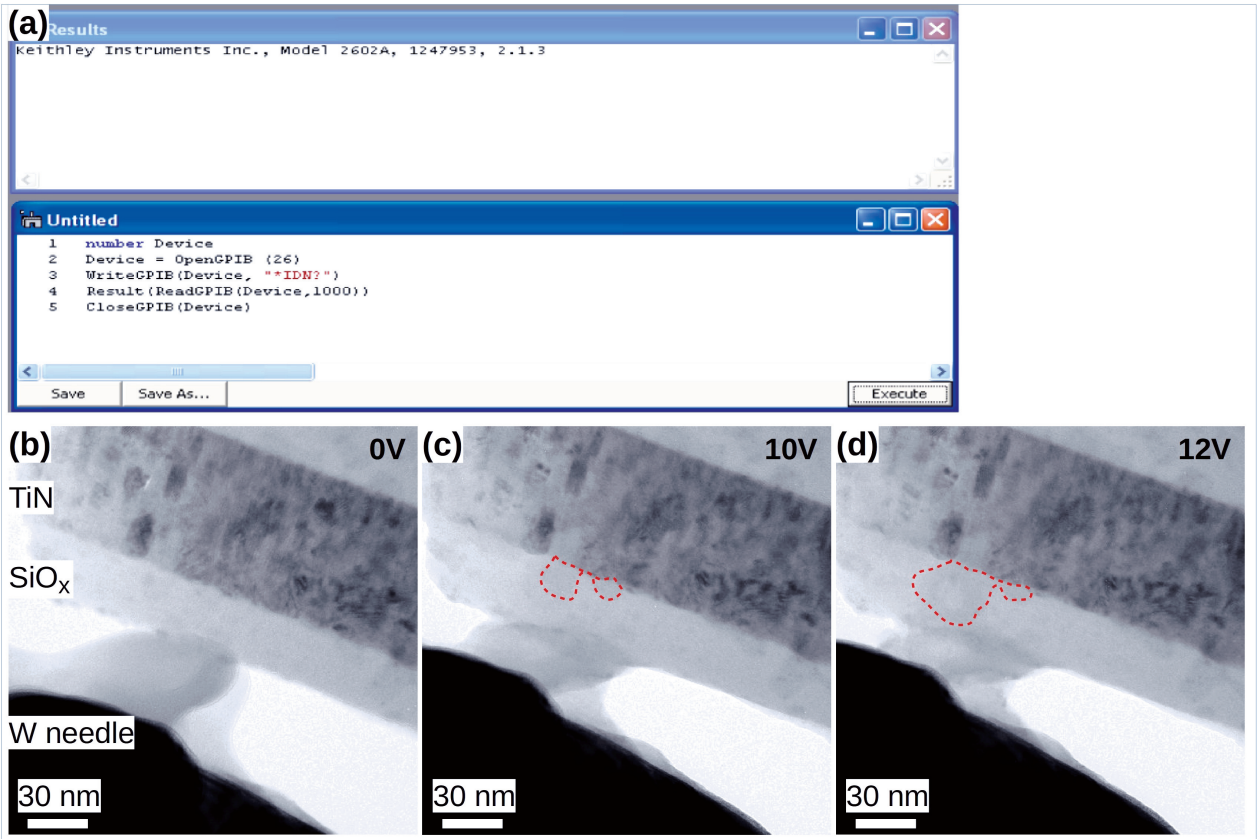

Figure 1: (a) Screenshot showing part of a Digital Micrograph script used to send and record electrical information to and from a Source-Measurement Unit. (b-d) Bright-field TEM images extracted from movies of a silicon-oxide-based resistive switching device biased in situ in the TEM using a movable W needle. The red dashed lines show changes in contrast observed during in situ TEM switching.

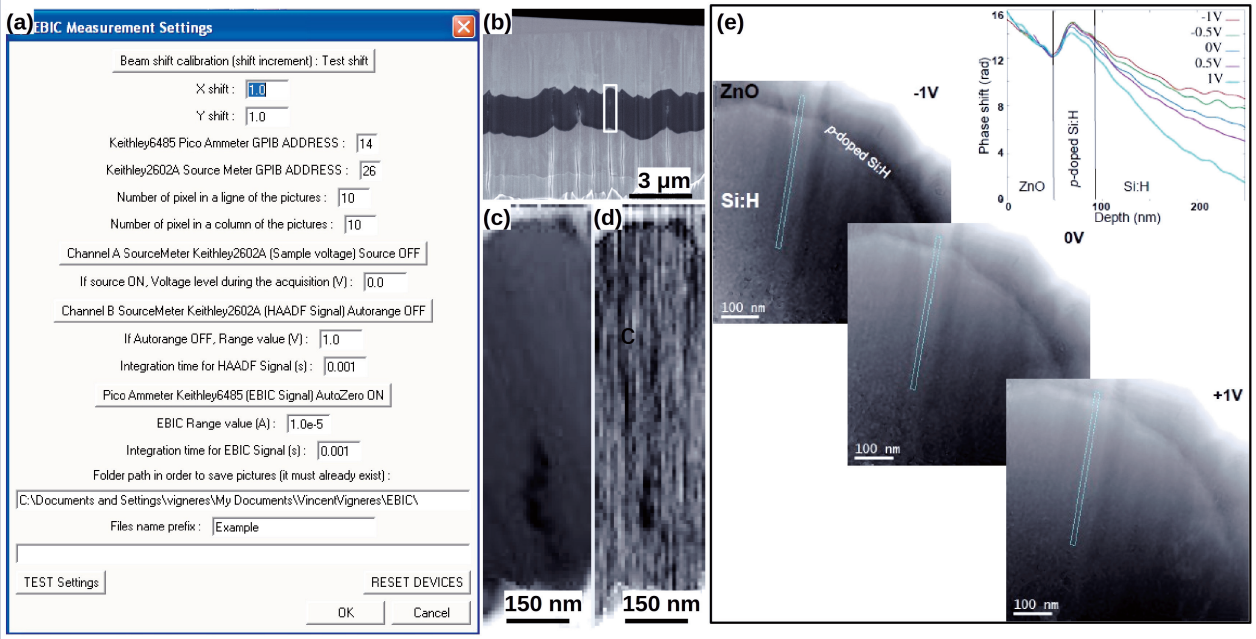

Figure 2: (a) User interface of a DM script used to record an EBIC signal in the TEM. (b) SEM image of a µc-Si:H solar cell. The white rectangle in (b) indicates the region from which the (c) STEM and (d) EBIC maps were recorded using the script shown in (a). (e) Phase shift images reconstructed from a series of off-axis electron holograms recorded automatically with applied voltages of between -1 V and +1 V. A vacuum reference electron hologram was recorded between the acquisition of successive pairs of electron holograms of the specimen.