Focused ion beam specimen preparation for electron holography of electrically biased thin film solar cells

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Thin films of hydrogenated Si (Si:H) can be used as active absorber layers in solar cells deposited on low cost substrates using plasma-enhanced chemical vapour deposition [1]. In order to improve the efficiencies of such solar cells, the nature of the defects that act as charge recombination centres and decrease the internal electric field in the active Si layer needs to be understood. Chemical analysis using transmission electron microscopy (TEM) reveals diffusion of ZnO and/or B from the top layers into the active Si layer [2, 3]. Measurements of bulk electrical properties suggest that the diffusion of dopants creates a conductive path between the two electrical contacts of the cell, resulting in a short circuit and degrading the carrier collection and overall cell efficiency. The carrier separation processes in thin film Si:H cells are driven both by diffusion and by the internal electrical field generated by the \sim 20 nm-thick *n* and *p*-doped Si:H layers that are deposited on both sides of the ~2 µm-thick intrinsic Si layer. In order to understand the recombination mechanism and the internal electric field in such an *n-i-p* stack, direct observation of these two quantities at the nanometre scale is needed. Carrier collection can be mapped with the required spatial resolution by recording the electron beam induced current (EBIC) signal, while the internal electric field can be measured using off-axis electron holography [4]. Previous attempts to measure internal electrical fields in solar cells have been limited by poor signal-to-noise ratio and difficulties in interpreting measured holograms. These limitations can be overcome by measuring the electron holographic phase shift as a function of applied electrical bias in situ in the TEM. An EBIC signal can then also be measured, either at 0V or in the presence of a forward or reverse bias.

One of the major limitations for the preparation of electrically biased TEM specimens of solar cells is the lack of a reliable and routine procedure that provides both high specimen quality (with few defects introduced by TEM specimen preparation) and high conductivity of the electrical contacts [5]. Here, we propose a fully focused ion beam (FIB) based approach that can be used to prepare a TEM specimen for electron holography of a thin film solar cell using conventional lift-out specimen preparation and a home-made MEMS chip. Our procedure allows an electrically biased TEM specimen to be prepared in less than a day with a high success rate. The novelty of our approach results from the use of: (1) an Omniprobe manipulator to transfer the specimen from the bulk solar cell to the homemade MEMS chip; (2) an FEI Omniprobe grid support to handle the MEMS chip; (3) ion-beam-deposited Pt to achieve low resistance electrical contacts without damaging the region of interest; (4) back-side ion milling to obtain a specimen that is suitable for electron holography.

Figure 1a shows the central part of a home-made MEMS support, onto which a TEM specimen of a solar cell has been attached using two ion-beam deposited contacts. Figure 1b shows a FIB-prepared solar cell ready for electron hologram acquisition. The use of back-side milling has minimised any curtaining effects that would have resulted from uneven milling due to defective regions in the solar cell, which is now only present on the top contact. The final electrically biased TEM specimens showed a strong decrease in electrical resistance from 2.4 to $1k\Omega$ when the ion energy was decreased from 8 to 2 kV for the final cleaning step.

Preliminary electron holography experiments were performed in Lorentz mode in a Titan TEM equipped with an image C_s corrector and a double biprism. For the present experiments, a voltage of 80 V was applied to the upper biprism. An electron hologram acquired from the intrinsic Si region in the solar cell is shown in Fig. 2a. The interference fringes in the FIB-prepared specimen have sufficient contrast to reconstruct the amplitude and phase, as shown Figs 2b and c, respectively. The present specimen preparation technique is applicable for *in situ* electrical biasing of any region of interest in a bulk sample, for both EBIC and electron holography studies. Furthermore, the use of FIB milling allows a wide variety of specimen geometries to be fabricated.

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Figure 1. (a) Scanning electron micrograph of the central part of a home-made MEMS support, on which a TEM specimen of a solar cell is attached. (b) FIB-prepared TEM lamella of a solar cell ready for TEM examination. The ion beam milling direction is opposite to the growth direction of the sample (i.e., back-side milling)



Figure 2. (a) Off-axis electron hologram recorded from the specimen shown in Fig. 1, which is in a geometry suitable for *in situ* electrical biasing. (b) Amplitude and (c) unwrapped phase images reconstructed from the hologram shown in (a). Arrows (1) and (2) indicate regions from which the 50-pixel-wide line profiles show in Fig. 3 were obtained. The white arrow indicates the growth direction of the solar cell.



Figure 3. Linescans of the phase shift in radians, obtained from the phase image of the solar cell shown in Fig. 2c.