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Site-specific TEM specimens from low contrast materials with nanometre precision

As it is known acquiring high quality data from transmission electron microscopes (TEMs) demands specimens with remarkable quality. This aspect is even more sensitive in the new generation of C_s corrected TEMs. In order to extract nanoscale devices and specific microstructural features with a nanoscale precision the combined focused ion beam/scanning electron microscope (FIB/SEM) is used. While this technique is very handy and has been used in different fields for many years it has its own limitations. Specially preparing site-specific specimens from nanostructures (<100 nm), such as nanoelectronic devices and samples made of very low contrast materials are very challenging. In low contrast materials it is very difficult to find a reference mark as a sign to stop milling or for drift compensation. A promising solution to solve these problems is using location marks. Different methods based on using X-shaped marks have been proposed. Due to the fact that in all of these methods the location marks are positioned outside of the lamella zone and the specimens are thinned down as much as possible before doing the lift-out procedure, they are not suitable for extraction of nm-size electronic devices and low contrast materials.

We developed a site-specific *in situ* lift-out method based on the regular lift-out technique, where the location marks are within the lamella zone (Fig. 1) [1]. These marks are filled with Pt and hence have a high contrast and can be used as milling end point also for the final thinning. The extraction was carried out using a FEI Strata 235 Dual Beam equipped with gas injection systems, for the possibility of depositing Pt and W, and an Omniprobe needle for *in situ* lift-out work.

Fig. 1. Schematic of location marks on both sides of the area of interest that is covered by a Pt layer [1]

Here we explain the method for extraction of a 400 nm wide $Al/AlO_x/Al$ tunnel junction used in superconducting quantum devices. The first step was to locate the area of interest (AOI) using SEM (Fig. 2a). A protective Pt-layer with the dimension of 5, 1, and 0.2 µm was subsequently deposited (Fig. 2b) over the AOI by electron beam assisted deposition (EBAD). In the next step, three location marks with dimensions of 0.1, 0.4, 1 µm, 0.1, 1.0, 1 µm and

0.1, 2.0, 1 µm were milled on each side of the junction of interest (Fig. 2c). Thereafter, a thick protective layer of Pt was deposited on top 15, 3 and 1.5 µm using ion beam assisted deposition (IBAD). At this stage the marks were thus filled with Pt and the regular lift-out procedure could proceed. After the trenches were milled on both sides of the Pt cover, the specimen bar was cut loose underneath and on one side. The Omniprobe needle was inserted and soldered to the bar using Pt deposition (Fig. 2d &e). The remaining anchoring strip was removed using ion milling and the bar was free to be extracted by the Omniprobe needle. The rest of the sample was lowered and removed. The next step was to insert the TEM grid and attach the bar to the grid using Pt deposition. Afterwards, the Omniprobe needle was cut free and retracted.

The final thinning of the bar was performed as follows; a high current ion beam (1000 pA) was used to mill the front side of the bar until the first marks were visible. Then the backside was milled until a bar thickness of 2 µm of the specimen was reached. At this stage the current was lowered to 300 pA and milling was continued on the front side until the next pair of marks were visible. The backside was then again milled until a thickness of 1 µm of the specimen was reached. Thereafter, the bar was milled by 100 pA current until the two last marks became visible (Fig. 2f). The milling of the backside continued until electron transparency was achieved. Fig. 2g shows the two location marks that are closest to the AOI. In order to reduce the surface damage the specimen was milled using low kV Ar ion milling (0.7 kV). Fig. 3 shows the result of high resolution TEM of the Al/AlO_x/Al tunnel junction specimen, where a nm-size junction was successfully extracted.

We have evaluated this method for two other types of structures as well. An artificial grain boundary junctions of high-T_c $YBa_2Cu_3O_{7-\delta}$ (YBCO) and a WC-Co based cemented carbide with Cr additions (bulk material), where there was an interest in analysing a binder phase grain boundary, represented other specimens with a need for site specific extraction of TEM specimens with high spatial precision. The extractions using the above described protol have proven to be reproducible and have high rate of success.

[1] Henrik Pettersson, Samira Nik, Jonathan Weidow, and Eva Olsson, A Method for Producing Site-Specific TEM Specimens from Low Contrast Materials with Nanometer Precision, Microsc. Microanal. 19, 73–78, 2013

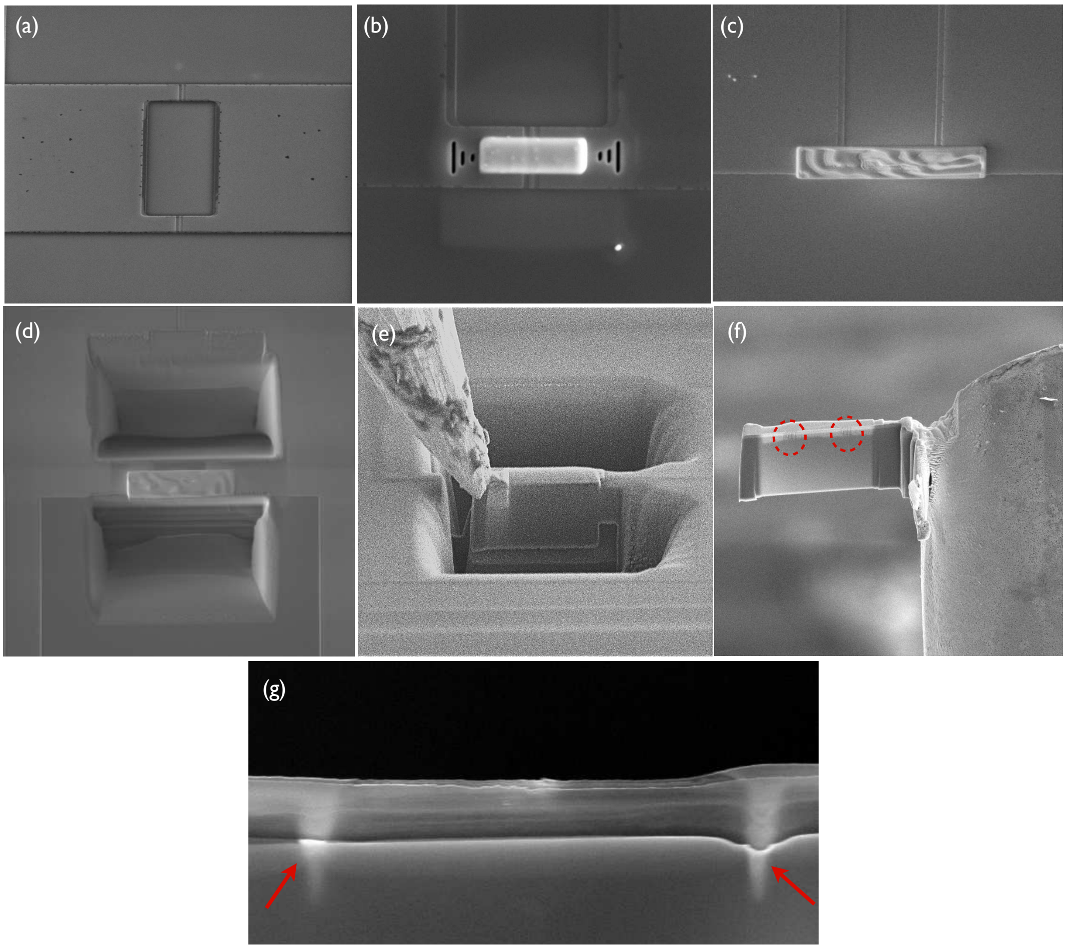

Fig. 2. The SEM images from the FIB/SEM showing the in situ lift-out procedure for an Al/AlO_x/Al junction. The location marks are highlighted by dashed circles in (f) and the two closest to the AIO are pointed out in (g).

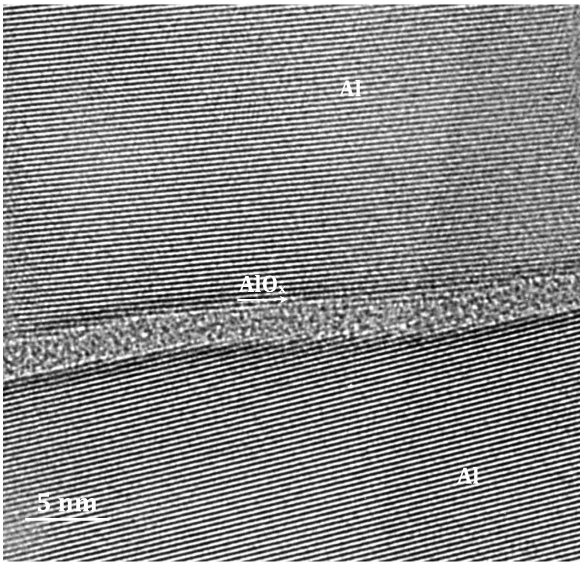

Fig. 3. High-resolution TEM image of the desired Al/AlO_x/Al junction