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Enabling Science and Technology through European Electron Microscopy

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Deliverable 6.2

Report on protocols and standards developed in ESTEEM2

Deliverable leader 6 – Max Planck Institute for Intelligent Systems, Stuttgart

FIB sample preparation of VLS ITO nanowires grown epitaxially on YSZ

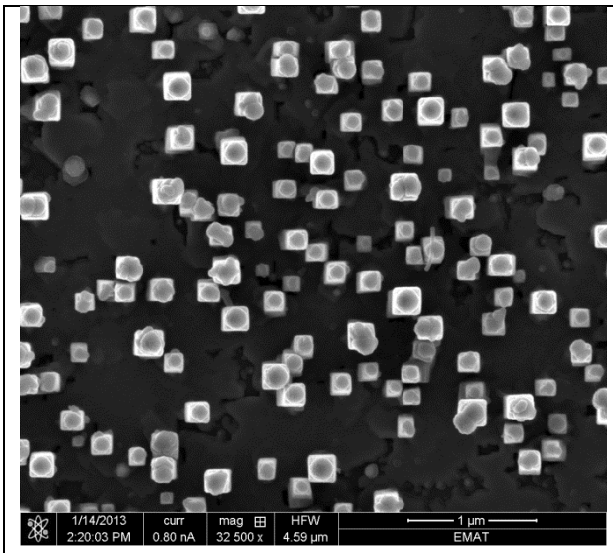
Semiconductor nanowires are of huge interest to the scientific community, as they can be applied in many fields like photonics, energy harvesting, electronics and gas sensing.

In order for this type of wires to be incorporated into layered devices, epitaxial growth of semiconductor nanowires on a substrate needs to be achieved in a controlled fashion. Also, an atomic resolution characterization of the nanowire-substrate interface needs to be performed, as the wire-substrate interface structure is of crucial importance for the device properties.

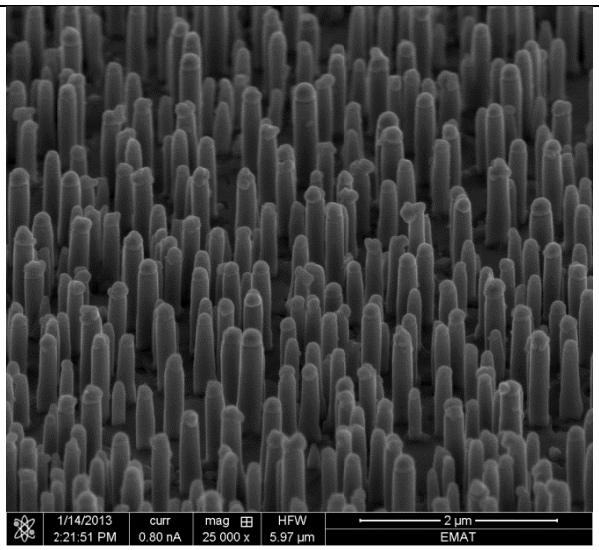
In this protocol, we describe the focused ion beam (FIB) sample preparation of epitaxially grown Vapor-Liquid Synthesis (VLS) Indium-Tin Oxide (ITO) nanowires on Yttrium-stabilized Zirconia (YSZ) substrates. This study requires intact nanowires to be cut out while still epitaxially attached to the substrate, in order to be able to study the atomic structure at the interface.

The focused ion beam instrument used for this protocol was a FEI Helios FIB-SEM, and following sample preparation steps were carried out;

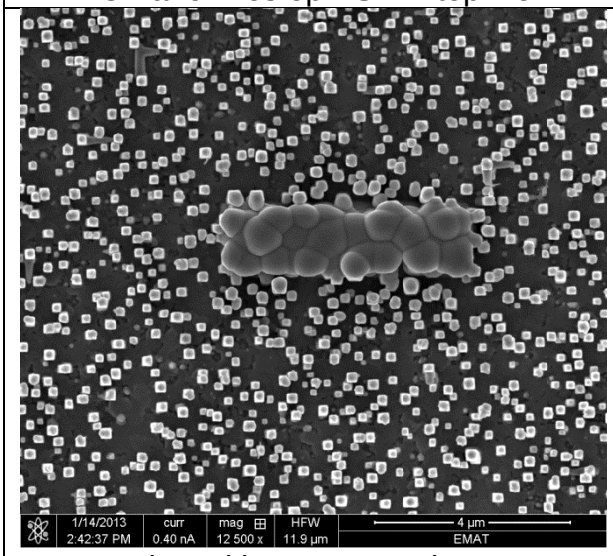
1. Careful selection of a row of epitaxial nanowires, to increase the change of an intact interface in the final sample.
2. Coating of the nanowire row using electron beam deposited Pt (e^--Pt, fine grains, approximately 1 micrometer thick layer, gentle deposition) followed by ion beam deposited Pt (Pt, coarser grains, approximately 2-3 micrometers thick).
3. Milling of two pits at either side of the nanowire row using Ga^+ ions at 30 kV beam energy at 9nA beam current.
4. Undercutting of the sample on one side, mounting of the omniprobe for sample lift-out, followed by the final cutting and release of the sample.
5. After lift-out, the sample is mounted in a two beam geometry onto a pre-milled Cu support omniprobe grid.
6. Gentle thinning of the sample for electron transparency, using 30kV Ga^+ ions for initial thinning, 8 kV Ga^+ ions for finer thinning and 2 kV Ga^+ ions for final thinning and sample cleaning.



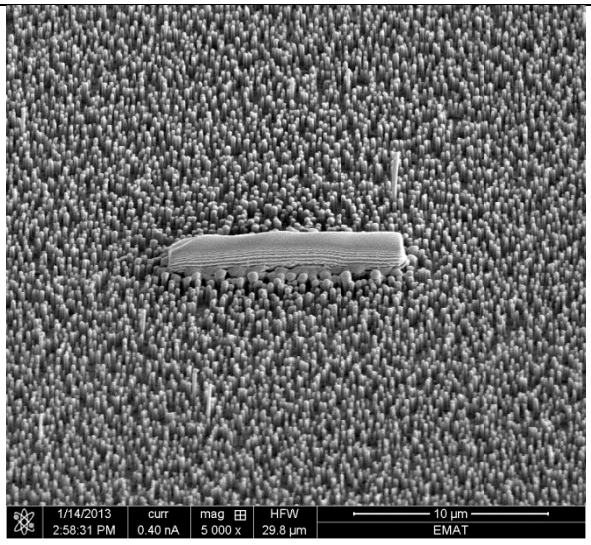
ITO nanowires op YSZ in top-view



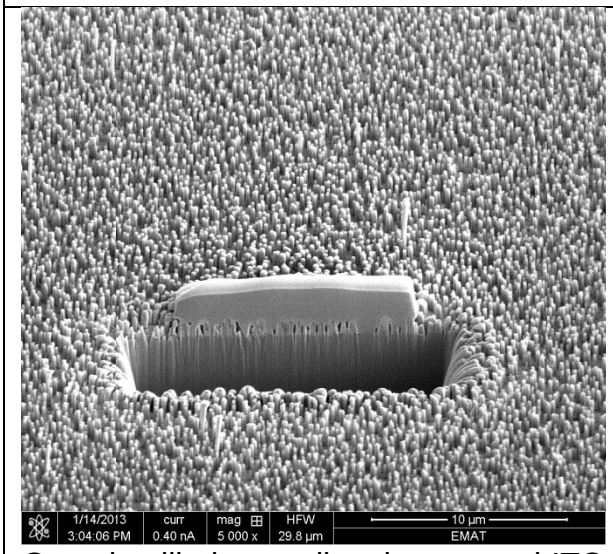
ITO nanowires in tilted view



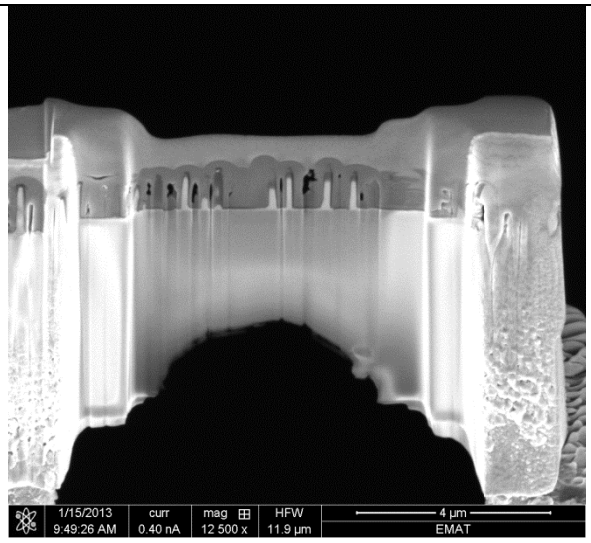
Pt deposition on nanowire row



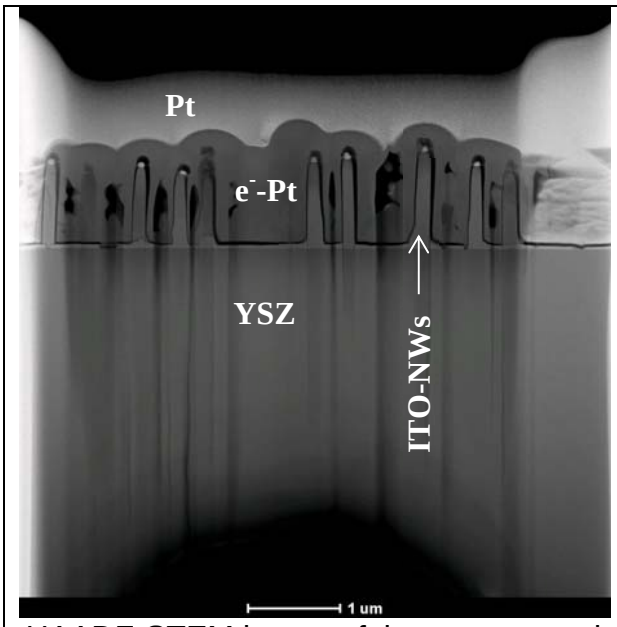
Pt deposition on nanowire row in tilted view



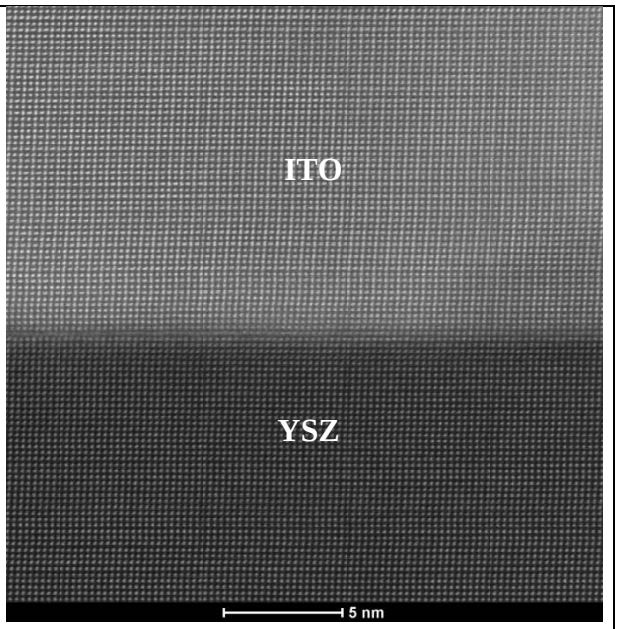
One pit milled, revealing the covered ITO nanowires



After lift-out and thinning, revealing epitaxial ITO nanowires on the YSZ substrate.



HAADF-STEM image of the as-prepared FIB sample, showing intact ITO nanowires grown in the YSZ substrate



HR-HAADF-STEM image of the ITO NW (top) – YSZ substrate (bottom) interface after sample preparation by FIB.