

## “Stand-Out”: A Novel Approach for Preparing Sub-100 nm Samples Through *in situ* Ion Induced Bending

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High resolution imaging techniques, such as Transmission electron microscopy (TEM) and Atom Probe Tomography (APT), are powerful tools for both materials and biological science. The success of imaging, however, rely heavily on sample preparation methods posing several challenges. Samples prepared for TEM need to be electron transparent (50-100 nm). Focused ion beam (FIB) has been a popular choice for site-specific TEM sample from bulk specimens and *in-situ* lift-out using a micromanipulator [1], [2][3]. This method, however, is time consuming and requires significant efforts and operator skills.

In this work, we introduce “*stand-out*”, a novel sample preparation method capitalizing *in-situ* ion induced controlled bending for TEM applications. Gallium FIB is a dual beam FEI Quanta 3D system was used for fabrication of suspended nanowires (NWs), which were subsequently manipulated controllably through ion irradiation induced bending [4] to *stand-out* until becoming vertical to the sample surface. The method is shown step-by-step through schematics and scanning electron microscopic (SEM) images (Fig. 1). Trapezoidal trenches on the planar surface are milled to obtain a thin specimen wall, which is further used to obtain the suspended NW (100-200 nm) employing FIB under-milling (Fig. 1a). This suspended NW is cut at one end to enable the bending through controlled dose ion irradiation at the other end (Fig. 1b). The NW gradually aligns towards incident ion beam with increasing ion dose. The implanted-ions, generation of point defects, and dislocated lattice atoms contributes to the local development of stresses and ion irradiation induced plastic deformation. Further, annular milling is employed to thin the NW to enable the electron transparency required for TEM imaging (Fig. 1c). The capability of the proposed methodology is demonstrated through TEM sample preparation for different materials such as Silicon (Si), Tungsten (W), Molybdenum (Mo). A TEM image of a prepared Mo sample is presented (Fig. 2). Additionally, the developed *stand-out* method has the potential for atom probe tomography (APT) sample preparation in the form of a sharp needle with an end radius 50-100 nm without requiring the lift-out (Fig. 2). Thus providing a unique route for correlative imaging with TEM and APT yielding structural and chemical composition not possible previously [4]. In summary, the successful demonstration of *stand-out*, a novel sample preparation method encompassing different materials through *in-situ* ion induced bending in a dual beam FIB-SEM microscope provides a novel approach with a huge potential for TEM/APT applications even in a cryogenic environment [5].

### References :

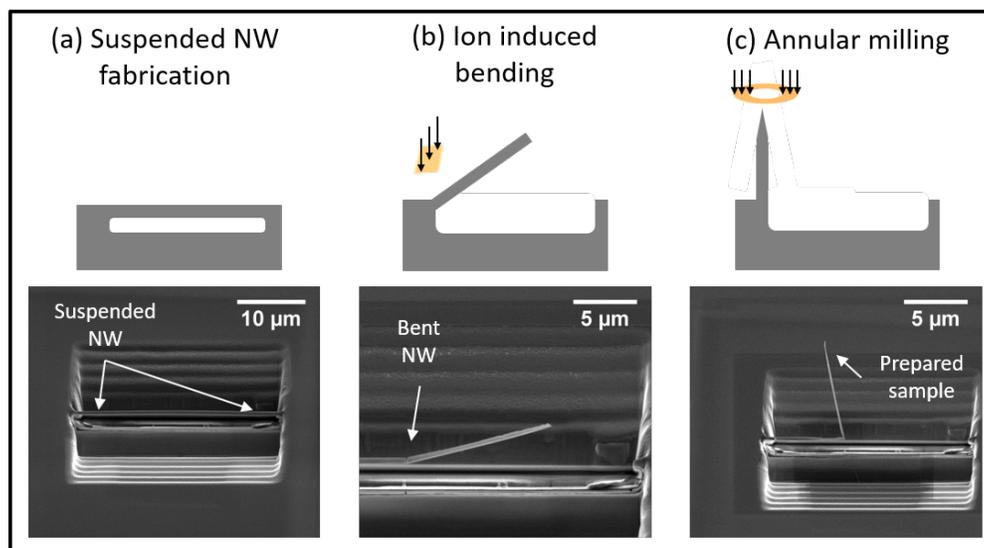
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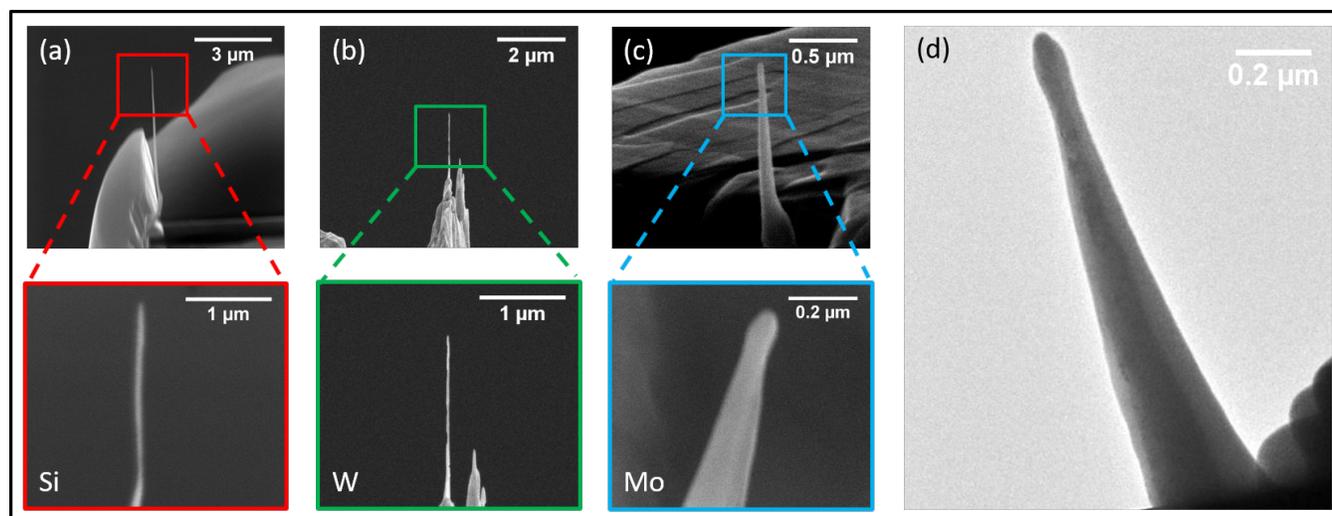
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**Figure 1.** Step-by-step illustration of *Stand-out* sample preparation method for TEM/APT in a dual beam FIB-SEM microscope: schematic diagrams and SEM images showing (a) fabrication of suspended nanowire (NW), (b) ion induced bending for vertical alignment, (c) annular milling for final polishing



**Figure 2.** SEM images of samples prepared on different materials using *stand-out* method in a dual beam FIB-SEM microscope: sub 100 nm sample of (a) Si, (b) W, (c) Mo. (d) TEM image of a Mo sample prepared by the “stand-out” approach.