In-situ tensile straining for two dimensional nanomaterials in the transmission electron microscope

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The experimental studies of the mechanical properties of nanomaterials have become a very important and challenging field of research in nanoscience, which frequently revealed new properties that have never been observed in bulk materials. The discoveries give promising technological applications and also stimulate scientific discussion among scientists. Many characterisation techniques in the electron microscope have been developed to gain fundamental knowledge of materials. The use of the picoindenter[1], the bimetal technique[2] and the atomic force microscope-transmission electron microscope (AFM-TEM) holder[3] in the TEM have become major techniques. These techniques are used to study the strength of nanomaterials and atomic-scale deformation mechanisms during straining. However, these techniques possess a great difficulty in the preparation or could potentially cause some contamination problems in TEM samples.

We have developed a homemade sample stage (shown in Figure 1) for tensile testing in TEM for two dimensional (2D) materials. This relatively simple tensile stage is made of a copper sheet with a thickness of 100 mm. There are three holes on it, two holes are for the screws of the TEM straining holder and a middle hole is for a thin holey foil where the 2D sample will be located. The 2D nanomaterial (e.g., BN) from a solution will be dropped on the thin foil. The following sample fabrication to cut a dog bone shape was performed by using the focused ion-beam (FIB) technique. After that, in-situ tensile testing was performed in TEM. This homemade sample stage could not obtain the magnitude of the tensile force but it is able to observe the deformation processes. The only disadvantage is that the straining holder for the sample stage is a single tilt. Therefore, it is challenging to obtain an accurate orientation for in-situ high-resolution imaging in during the straining.

To overcome the tilting problem, part of the stage that has a sample can be cut and attached to a commercial TEM grid for further analysis using a double tilt holder after the in-situ testing. The main advantage of this tensile stage is that the tensile direction can be adjusted to the orientation of the sample prior to the process in FIB. This will allow us to choose a certain tensile orientation with respect to a crystallographic orientation of the material. Our homemade sample stage has successfully used for some 2D materials (shown in Figure 2) including graphene, Boron Nitride and WS_2 and to observe the dynamic deformation processes until fracture of the materials.

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References

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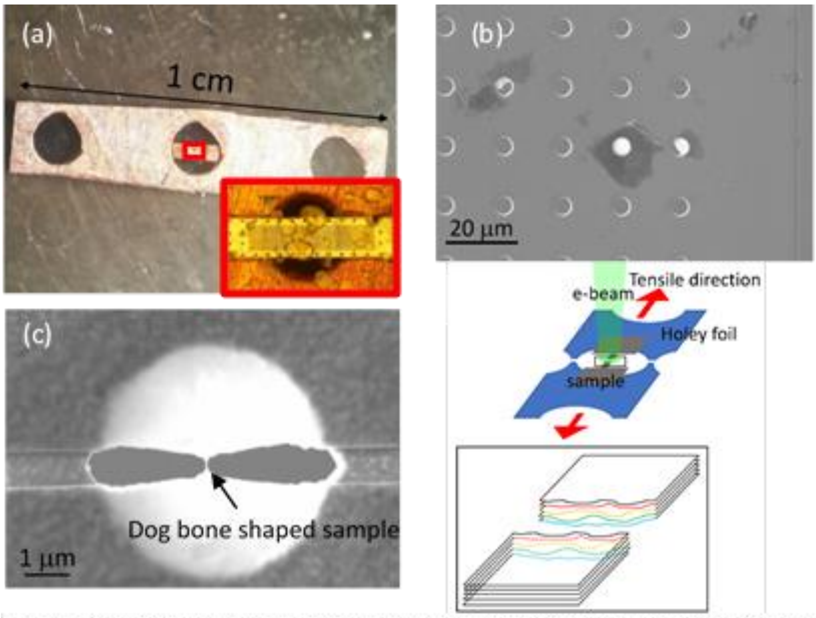

Figure 2 (a) A homemade sample stage to perform tensile test for two dimensional nanomaterials in TEM. Inset: thin foil attached on a smaller copper bridge. (b) A flake of sample indicated by an arrow covers a hole on the thin foil. (c) A dog-bone-shaped sample prepared by FIB. (d) Schematic of a tensile experiment process in TEM.

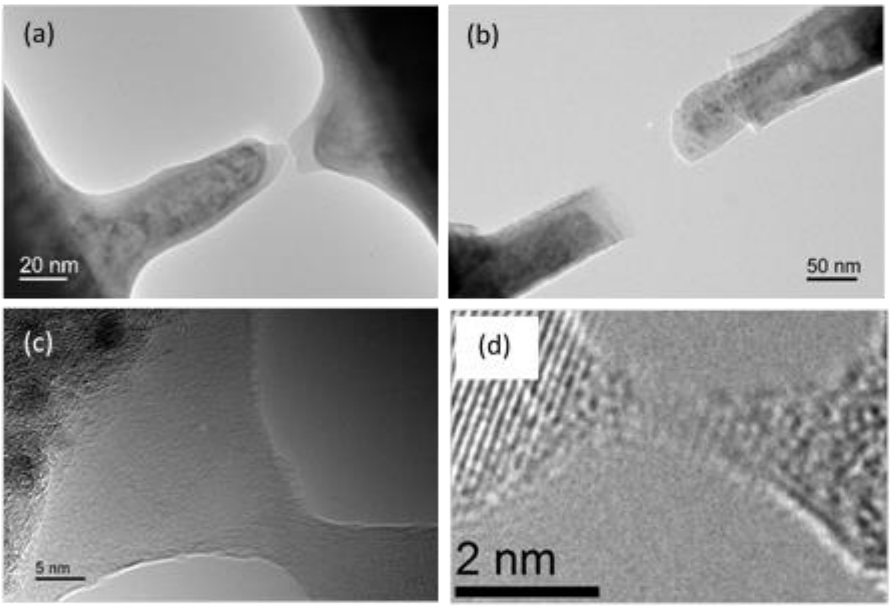

Figure 2 (a) and (b) A sample after being pulled to fracture using this sample stage. (c) and (d) A deformation process at high magnification observed during the ductile mode.