Characterising ambient and vacuum performance of a miniaturised TEM nanoindenter for \textit{in-situ} material deformation

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Abstract. A miniaturised nanomanipulation and nanoindentation system has been designed and manufactured to perform sub-micron localised \textit{in-situ} deformation studies in a high resolution transmission electron microscope (HRTEM). The nanomanipulation drive comprises two independent mechanisms for both coarse and fine positioning of sharp indenter tips. Small slip-stick inertial sliders are used to coarsely position a tip which fits inside a bespoke hollowed specimen holder for a JEOL (Japan) 2010/3010 series microscope. The coarse drive comprises three fully independent sliders which are set mutually perpendicular to one another ($x$, $y$ and $z$) with a range $> 1$ mm and resolution $\sim$ 100 nm. Fine positioning is achieved with a quartered piezoelectric tube with range $\sim$ 2 µm and resolution $< 1$ Å. Optical displacement sensors have been used to characterise the nanomanipulation drive performance including total displacement rate and step size in ambient conditions. These are compared to the operation of the drive within a TEM under vacuum conditions. TEM observations at high magnification enable optimisation of the fine and coarse motion and overall drive stiffness.

1. Introduction

\textit{In-situ} TEM nanoindentation technology has been developed over the past decade and has recently attracted significant interest because of its great potential in quantifying the mechanical and tribological behaviour of a broad range of nanostructured material systems. [1-4]

Combining nanoindentation with TEM can realise \textit{in-situ} dynamical real-time mechanical testing of nanostructures. However, there are a number of significant technical challenges for the realisation of nanomanipulation and nanoindentation drives including spatial limitation, high vacuum and 3-dimensional positioning requirements in the range of mm $\rightarrow$ Å. In this paper, a miniaturised nanomanipulation and nanoindentation system for a high resolution transmission electron microscope (HRTEM) will be presented and the performance in ambient and vacuum is demonstrated and discussed.

2. Nanoindenter design

A miniaturised nanomanipulation and nanoindentation system has been designed and manufactured to fit inside a hollowed specimen holder for a JEOL 2010/3010 (JEOL, Japan) series microscope. Figure 1 shows a photograph of the assembled nanomanipulation drive inside the TEM holder. The size of the drive has been miniaturised down to less than 8mm in diameter.
The nanomanipulation and nanoindentation drive consists of two independent mechanisms for both coarse and fine positioning of a sharp indenter tip. Inertial sliders, which have been used in many fields because of their compactness, stability, precision and flexibility of control, are used for coarse positioning with three fully independent sliders to realise three-Cartesian-axis (x, y and z) displacements with a range >1 mm and step resolution ~ 100 nm.

The fine x, y and z motions of the tip are generated by a four-quadrant piezoelectric tube (EBL, USA) with 0.125” diameter and 1” length. The piezoelectric tube has a maximum displacement of ~2.3 μm along the long direction (z axis) of the holder and ~ 18 μm in the other two perpendicular directions when ± 100 V is applied.

3. Performance characteristics of the nanoindenter drive

3.1. Ambient and vacuum performance of coarse positioning drive.

In order to evaluate the nanomanipulation and nanoindenter on an optical bench, three fibre optic displacement sensors (D63, Philtec Inc. USA) have been used in ambient conditions to monitor the motions in three Cartesian axes. The stability and repeatability of motion of the inertial slider is essential for the coarse drive to approach the tip to the area of interest. Typical time-displacement curves of the x inertial slider are shown in Figure 2a. The steps in the curves come from intervals between groups of exponential waveform pulses and a displacement rate of ~ 40 μm/s is appropriate for the coarse motion. The two groups of six sequential displacement curves (A and B) show very good repeatability in both opposing directions. The x and z drives in the horizontal plane have very similar performances and similar time-displacement curves have been measured in ambient conditions.

Over 3 mm movement has been achieved in the z direction and 1.5 mm in the x direction. The motion of the vertical y-drive is complex due to the influence of gravity, and is discussed in another paper [5].

To evaluate coarse motion at high resolution, the drive was put into a JEOL 2010 TEM and a tungsten tip was mounted which can be observed in the TEM at high magnification. All motions of the tip apex due to both coarse and fine piezoelectric actuators can be recorded on video at 25-frames/second. Figure 2b shows the coarse movement in the x-z plane obtained in the TEM. The motions along the xyz axes were independent of one from another. Tracing the motion of tip apex along x or z-axis, allows the actual projected angle between the two axes to be measured which is ~84º for this nanomanipulation and nanoindentation system. Note that the z motion is not parallel with axis of the tip end in Figure 2b because the tip end was bent.

The in-situ movement of the x coarse slip-stick piezoelectric plate was observed in the TEM at very high resolution. The track of tip motion can be measured by analysing the continuous video frame sequences and an example is shown in Figure 3a. Elastic recovery of the piezo and support after each step is clearly revealed. The time-displacement curve of the inertial slider for an applied exponential waveform pulse is shown in Figure 3b. A resultant ~ 125 nm step size in the x direction can be measured from Figure 3b. The step size could be set down to ~ 100 nm by changing the normal force applied on the slip-stick slider.
3.2. Ambient and vacuum performance of fine piezoelectric tube.

Optical bench testing has been carried out using fiber optic displacement sensors for evaluation of the fine piezoelectric tube. The displacement in $x$-axis is shown in Figure 4a. Figure 4b shows six displacement curves obtained in $y$ direction and reveals very good repeatability in different measurements. The fine piezoelectric tube has similar performance with total movement range about 35 μm and 30 μm at tip end respectively in $x$ and $y$ axes when ±100 V voltage is applied. The $z$ motion in opposing directions is shown in Figure 4c and ~ 2.0 μm movement can be observed. The motions in $x$, $y$ and $z$ are not perfect linear because piezoelectric tubes exhibit hysteresis [6]. Therefore, there are asymmetric performances in the opposing directions in three-Cartesian-axes ($x$, $y$ and $z$).

It is difficult to do an accurate measurement of $z$ motion on an optical bench because of the ambient variations such as air flow, temperature and humidity which affect the test results in the sub-micron regime. Therefore, the motion of the fine piezoelectric drive also has to be evaluated by TEM observation. Less than 1 Å displacement can be achieved when a ~ 10 mV of voltage is applied. A $z$ displacement track of the fine piezo is demonstrated in Figure 5. TEM observation reveals the difference between the extending and retracting trace of the free tip before indentation. The hysteresis behaviour of the free tip should be recorded before indentation for quantifying force loading. The direction of motion due to the tube piezo is adjustable by changing an offset voltage applied on $x$ or $y$ electrodes of piezoelectric tube, enabling full $xyz$ control of the tip for experiments such as nanoindentation.

Figure 2. a) Typical displacement-time curves of $x$ inertial slider measured by using fibre optic displacement sensor A) right motion of the slider, B) left motion of the slider. b) Coarse movement along both $x$-$z$ axes observed in TEM to determine the projected angle between the two axes.

Figure 3. TEM observation of coarse drive motion. a) TEM image of a W-tip and the trace of tip end along the $x$-axis showing elastic recovery after each step. b) A time-displacement curve of the inertial slider motion seen in a).
4. Summary
A miniaturised 3-dimensional piezoelectric actuator system, which consists of two independent mechanisms for both coarse and fine positioning, has been constructed for in-situ TEM nanoindentation and nanomanipulation. Evaluation of performance of both the coarse and fine drives has been carried out in ambient and vacuum environments. TEM characterisation of the drivers at high magnification enables optimization of the drives and overall drive stiffness. The coarse drive comprises three independent sliders which are set mutually perpendicular to one another (x, y and z) with a range > 1 mm and resolution ~ 100 nm. With a few tens of microns per second speed, the coarse drives can quickly and accurately approach the tip to an area of interest. Fine positioning is achieved with a quartered piezoelectric tube with range ~ 20 µm and resolution < 1 Å along the x or y direction. With the fine positioning, 3 dimension nanoindentation and nanomanipulation functions can easily be realised down to sub-nanometer accuracy.

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