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A nanomanipulation system for tomographic examination of nanostructures on demand

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Abstract. In this paper, we present a recently upgraded 7-degree-of-freedom nanomanipulation system inside a TEM with focus on applications in electron tomography of nanostructures. Previously it has been emphasised that the traditional way of mounting of nanoparticles on flat carbon films might not be the best way to achieve very high tilt angle series free of missing wedge for tomography. Mounting of particles ex-situ at the end of nanotips allows for higher tilt but poses challenges in the success rate of getting the object attached to the very tip. Our mixed translation & rotation nanomanipulator based on 3 coarse positioners in x, y, z by piezoelectric slip-stick actuation and 3 fine directions by a piezo-tube, as well as a rotational piezodrive, allows not only to rotate a cylindrical object by up to a full circle, but now also features a second specimen mount for a “fixed sample”, which could host a reservoir of nanoparticles on carbon film, or nanoparticles inside nanopores. The translation system can then be used to pick up particles on demand and rotate them with the piezoelectric actuator. In particular, we describe how nanoparticles dispersed and transferred onto a normal TEM grid are picked up by a W tip for subsequent large tilt rotation.

1. Introduction

Electron tomography [1] is commonly used nowadays in materials research fields for the physical and chemical characterization of sub-micron sized materials with up to nanometer resolution in three dimensions. To reduce or eliminate missing wedge artifacts in tomography, methods include (i) wide angle single tilt (at best 180°) with special holders or TEM goniometers [2, 3] or (ii) double-axis tilt [4, 5], which is more challenging in alignment and higher in dose. Here we present a new nanomanipulation system [6] with 7 degrees of freedom inside a TEM with focus on applications in electron tomography of nanostructures.

The traditional way of mounting nanoparticles on a flat grid cannot achieve an 180° full tilt series due to the shadow effect of the grid even though the grid size can be smaller than the pole piece gap. Therefore, in order to avoid the missing wedge, it not only requires the holder/goniometer having the ability to rotate at least ±90°, but it also needs the objects not to be shadowed for a range more than 180°. The object could be mounted ex-situ at the end of a nanotip, but it is very challenging to succeed by chance mounting the object at the very end of the tip. Therefore we propose our 7-degree-of-freedom piezoelectric drive to lift out nanoparticles in-situ which can be controlled to attach to the end of the tip as much as possible to reduce the shadow effect from the tip, and a tomographic tilt series is collected afterwards for later tomographic reconstruction.
2. Materials and methods

The whole piezoelectric goniometer system [6] is composed of three sub devices: fine drive, coarse drive and the rotary drive. Three fine translational movements in X, Y and Z axes are achieved by a quartered piezo tube in the front of the whole system as the fine drive. In the middle of the system is the coarse drive containing three sliders which provide independent coarse translational movements along all three axes and is operated serially to the fine drive. At the back, the rotary drive is equipped with an angular sensor providing the rotational movement around the X-axis and accurate angular displacement feedback. The coarse X, Y, Z and fine x, y, z are independent coordinate systems, therefore we define them as different degrees of freedom, although strictly speaking in geometric terms, both coarse and fine drives move in a 3D Cartesian space. By adding the rotary drive, a rotary degree of freedom, seven degrees of freedom in total for the movements are achieved. Both the coarse drive and the rotary drive use a slip-stick inertial slider mechanism. For the rotary drive, the shaft on which the coarse and fine drives are mounted is centred inside a pair of piezos with opposite shearing directions along the Y-axis, and the rotation directions change accordingly when the polarity of the potential applied on the piezos is reversed. The translational drives are put ahead of the rotary drive on the specimen side, so that the specimen can be always centered after each angular movement. The whole piezoelectric goniometer is fitted inside a hollow standard specimen holder for JEOL 2000/3000 series microscopes, therefore there will be neither modification requirements to the TEM nor any compromise to the performance of the default TEM goniometer.

A tungsten tip is mounted on the fine drive to be used for picking up nanoparticles. A second specimen mount is fixed to the hollow holder shell, and can host a reservoir of nanoparticles on a cut grid, as shown in Figure 1 (a). The piezoelectric goniometer can work in parallel or in addition to the TEM goniometer, so we can use the TEM goniometer to move/rotate the grid to find interesting objects for lift-out. The W tip approaches the grid by using translational coarse and fine drive, then touches the desired particles and picks them up, shown in Fig 1 (b) and (c). The particle should be sitting at the end of the tip as much as possible to avoid the shadow effect for later rotation. Finally, the W tip with attached nano objects is retracted in a free space and rotated by the rotary drive for the tomographic experiment, which is shown in Fig 1 (d).

![Figure 1](image_url)

**Figure 1.** (a) A W tip is mounted on the fine drive of the piezoelectric system. A 3mm grid cut in half is mounted on the other side fixed to the holder shell as the reservoir of nanoparticles. (b) The tip approaches to the grid holding nanoparticles. (c) Choosing and lift-out of particles (purple) on the grid. (d) The tip is retracted and a tilt series is conducted.
It is not necessary that the nanotip is made of W. For instance a Ni tip might be more helpful to pick up magnetic nanoparticles, while Al and porous Al$_2$O$_3$ tips have been tested for high transparency in the past, or Au tips would be most oxide-free. The fixed sample reservoir is not necessary to be a cut copper grid either. It could be another nanotip, metal wire, piece of Si, etc., whatever is holding the nano-objects of interest. In this paper, we choose different grids as the reservoir. As to the nanoparticles, in theory any particles, subject to suitable size range, could be picked up. For demonstration in this case, we choose hydroxyapatite (HA) nanoparticles (courtesy of nano4med TSB consortium Nottingham/Sheffield) for their wide and important applications in bio-engineering [7], medicine [8], and drug delivery [9].

3. Experimental Applications
Tungsten tips were prepared and mounted as reported in [10]. BF-TEM tilt series of the W tip with attached HA particles were acquired using a JEM 3010 TEM (JEOL, Japan) at 300kV.

The first attempt was to disperse HA particles on an ordinary copper grid with holey carbon film (Agar), then cut the grid in half and mount it onto the fixed end of the actuation holder, as shown in Figure 1 (a). The W tip was moved by the coarse piezo drive, and the step size can be controlled from about 20nm to 150nm by using the minimum and maximum driving voltage. The W tip carefully approached the carbon film containing HA particles (in plate shape) and then touched a bundle of them, which is shown in Figure 2 (a). Due to Van der Waals force or charging induced electrostatic force of the W tip, a couple of HA plates were attached to the W tip, one was on top left and the other was on the right, as shown in Figure 2 (b). Now the one on the top could be used for tomography.

![Figure 2](image-url)

Figure 2. (a) The W tip is touching the carbon film containing HA platelets, scale bar is 200nm. (b) A couple of individual HA plate are picked up, scale bar is 200nm. (c) A piece of carbon film is lifted out by a W tip, scale bar is 500nm.

Not only particles, sometimes a piece of carbon film can also be lifted out by the W tip, which is shown in Figure 2 (c). The advantage of lifting out carbon film is that a reduction of the shadow effect is achieved as the objects on the film may be far away from the end of the tip. However, the size, shape and orientation of the carbon film fragment which can be picked up is totally uncontrollable, also the sticky carbon could crash/bend the tip before successful lift out. So a carbon film pick-up may not be very practical in reality.

When the HA nanoparticle size is very small, say less than 20nm, the W tip could not attract them from the thin film, as picking them up from a holey carbon film is equivalent to breaking the carbon film and lift out one piece of it. Therefore, for small HA particles, we dispersed them on a bare copper grid. The nanoparticle clusters, once loosely bonded on the copper wire, could then be easily broken and picked up by the W tip.
Figure 3 shows a lifted out HA particle cluster containing particles with size about 5nm on the top of a W tip. The clusters were rotated with the tip from 0° to 120° and images were taken with 5° intervals. A selection of images from this series is shown in Figure 3 with rotation angle at 0°, 25°, 50°, 75°, 100° and 120° respectively. Unfortunately the HA cluster was deformed by the irradiation damage, but from the combined shape of W tip and HA, a clear progress of tilt is still be seen.

Figure 3. A selection of images from a tilt series with 5° intervals from 0° to 120°. Scale bar is 50nm.

4. Conclusion
In summary, a newly developed 7-degree-of-freedom piezoelectric drive inside TEM can be used to choose and pick-up a nanoobject from a particle reservoir, and subsequently electron tomography can be achieved via unlimited tilt series, as demonstrated in [6]. Artifacts related to the missing wedges can be eliminated and the main benefit is that picked-up nanostructures are sitting on the top of the tip to avoid any shadow effect during the rotation.

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References