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Cone-Shaped Thickness Standard Made by Focused Ion Beam

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The thickness of the Transmission electron microscopy (TEM) sample is invaluable information. Sample thickness is usually obtained by indirect methods, such as electron energy loss spectroscopy (EELS) or convergent beam electron diffraction (CBED) [1]. To calibrate these indirect measurements, a thickness standard with well characterized thickness is desired.

Spherical particles have been used as thickness standards since they have directly measurable thickness [2,3]. However, a major disadvantage of spherical samples is their limited availability for different materials. This paper introduces a cone-shaped thickness standards (CSTS) as an alternative thickness calibration tool. The CSTS schematic is shown in Figure 1. The cone's rotational symmetry ensures that the thickness (*t*) through the center line of the needle is equal to the local width (*w*). There are two groups of measuring points along direction **a** or **b** in Figure 1. Those well-defined thickness variations are ideal for detailed thickness calibration.

A focused ion beam (FIB) with ring-pattern milling capability is an ideal tool for making a CSTS (Fig. 2). The material covered by the area between the outer ring and the inner ring is milled away to a certain depth. The inner ring controls the size and location of the needle. To reduce re-deposition, it is necessary to clean a relatively large area surrounding the desired location before ring-patterned milling. The ring-patterned milling creates a cone-shaped needle (or a conical frustum for a large inner ring) instead of a cylindrical column (Fig. 3). The cone shape is produced because the intensity distribution across the ion beam is not a delta function. The diminishing intensity at the edge of the ion beam controls the slope of the cone. The angle of the cone is a function of ion beam size or current [4]. Without sample drift, the rotational symmetry is ensured by lens control of the ion beam and the precision is expected to be high.

The deviation from a true cone can be examined through a cross-section view of the needle, or by tilting the needle along its axis in TEM. A CSTS made from Si with Pt deposition on the tip is shown in Fig. 4. The sample was tilted in TEM along the needle axis. The variation of the width was found to be less than 1% (Fig. 4B).

In TEM applications, it is critical to reduce thickness error from the unknown tilt of the CSTS needle. Two steps minimize the slanting of the needle: (1) The needle starting material is mounted onto a flat TEM half grid and the flat surface of the grid is aligned parallel to the ion beam in FIB viewed by ion beam. This step ensures that the CSTS is parallel to the grid. (2) In TEM applications, the CSTS is positioned at a right angle to the electron beam (the flat grid surface can be used as the reference during sample loading). With tilt error, the thickness of the CSTS deviates from $t = w$ to $t \approx w(1/\cos\theta + \tan\theta \times \tan\alpha)$ for small α, where θ is the angle of needle axis tilted away from horizontal plane and α is the slope angle of the cone surface. For example, the error of t is about 0.18% for $\theta = 2°$ and $\alpha = 2°$ and 0.73% for $\theta = 4°$ and $\alpha = 4°$. Thus, tilt-error from a small misalignment is negligible. It is useful to align the needle direction with a feature on the grid (such as a marker on the grid) before beginning the FIB process. By doing so, the CSTS can be loaded into TEM with a needle axis roughly parallel to the goniometer tilt.

The CSTS described in this paper is easy to make and has no material limitations. With a range of directly measurable thicknesses, it is a suitable tool for thickness calibration in TEM work.

References

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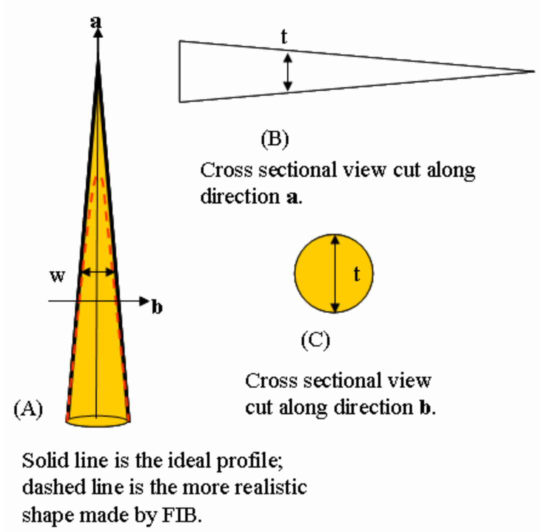

Fig. 1. Perspective view (A) and cross-section views (B and C) of the cone-shaped sample.

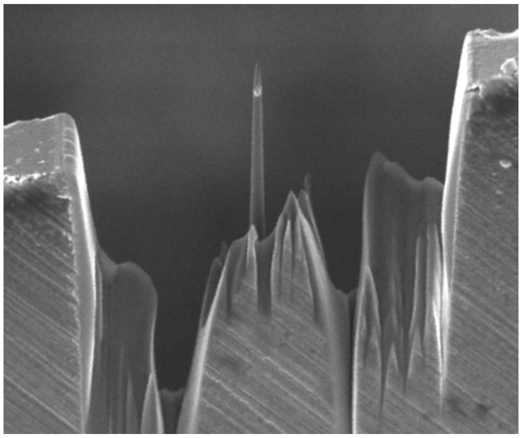

Fig. 3. SEM picture showing the cone-shaped needle formed from bulk material.

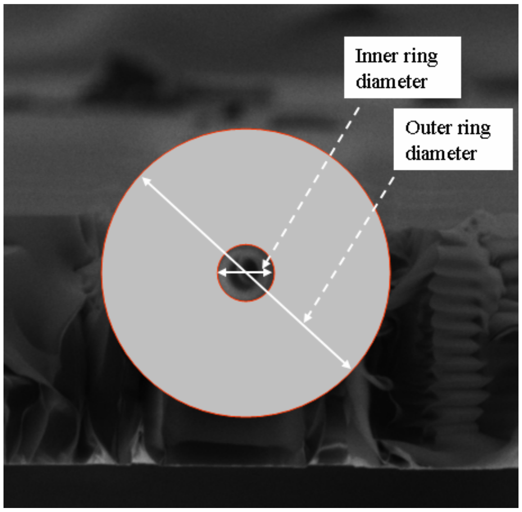

Fig. 2. Top-down view of bulk sample with the ring pattern on it. The inner ring controls the size and location of the cone.

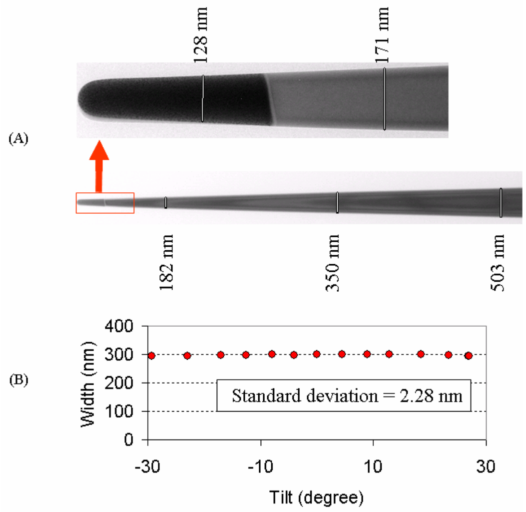

Fig. 4. (A) TEM images of a needle with some thickness labeled along the length. (B) The variation of a local width when the sample was tilted around the cone-axis.