

## Development of a New Type of Thin Film Phase Plate and its Application for In-Situ Observation

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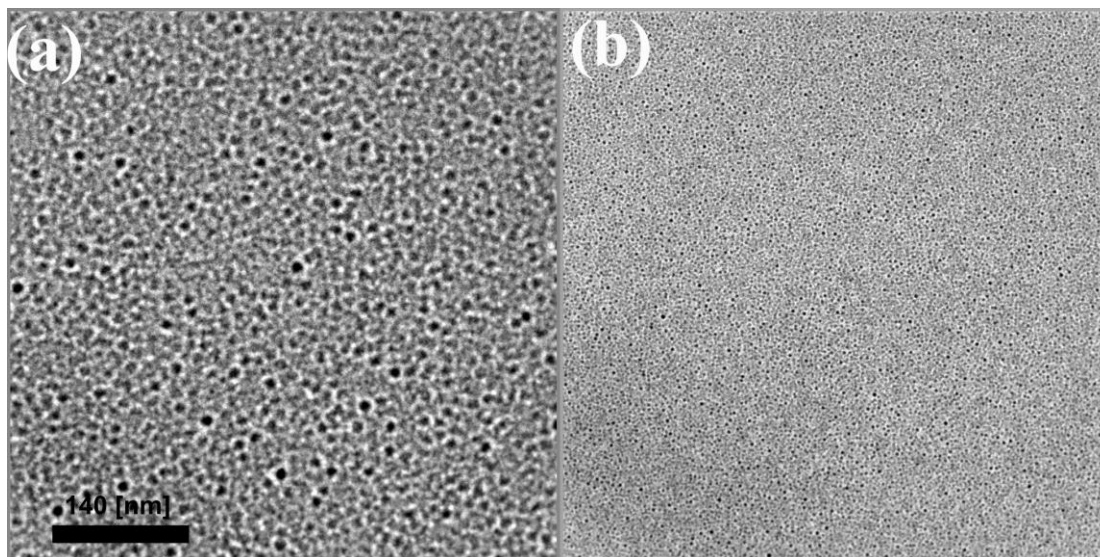
Biological molecules composed of lighter elements such as C, O, N, etc. do not provide strong interactions with imaging electron waves. They can be regarded as phase objects which only give phase shift to the electron waves. The biological molecules are very large comparing with single atom or single inorganic molecule. Thus, their imaging with the resolution of 1nm scale is very useful to discuss about their functions. However, a phase contrast transfer function (PCTF) of the objective lens in a commercial TEM gives very small values for a thing larger than 1nm, and the biological molecules cannot provide high contrast images by a conventional TEM.

The phase plate transmission electron microscopy (P-TEM) enhances the image contrast of a phase object with the size larger than about 1nm because the PCTF is modified by the phase plate. Several types of the phase plates have been developed. The Zernike phase plate made of a carbon thin film with a 1  $\mu\text{m}$  diameter hole at the center was the most effective to get application data until now. While the scattered electron waves pass through a film to be delayed their phase, unscattered waves pass thorough into the hole of the film. Thus, these waves interfere with each other to enhance phase contrast.

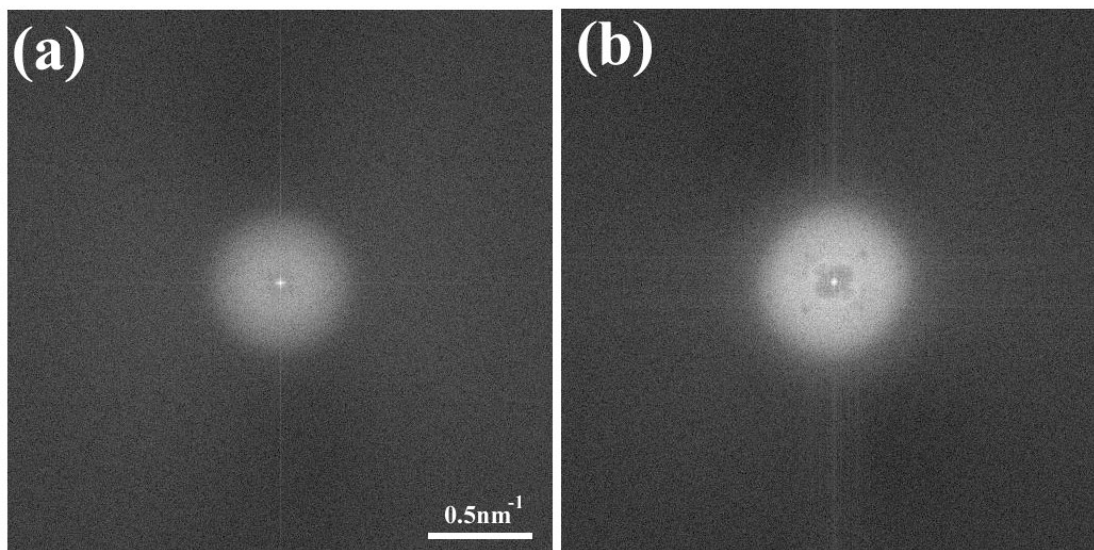
An amorphous carbon is the standard material for the Zernike phase plate and it provided many results from the beginning of the first success [1]. The carbon film phase plates have been improved but they are still unstable mainly due to charging effects. We have been developing an integration method of P-TEM and environmental TEM (E-TEM) for *in situ* observation of wet samples [2] and stability of the phase plates is one of the important factors for our aim. Recently, we started to develop a new type of phase plate. We used silicon nitride (SiN) membranes and dopant materials are injected to increase their conductivity. Since injection of the dopant was done by thermal diffusion by heating after the dopants deposition on SiN films, some of the deposited dopants form island if the annealing is not enough (Figure 1). The amorphous metals were deposited after the injection of the dopant to recover the flatness of the films. The deposition of the metal film increases the conductivity and a thin film phase plate which gives similar property to the carbon phase plate was obtained. Figure 2 shows a comparison of FFT patterns of the amorphous film images. Fig. 2(a) was obtained from a conventional TEM image and Fig. 2(b) was obtained from P-TEM image using a SiN based phase plate. In the present paper we will introduce the details of the new types of the phase plate and their application for *in situ* environmental phase plate TEM observations.

[1] Danev R. and Nagayama K., *J. Phys. Sci. Jpn.* **70**, (2001) 696-702.

[2] Inayoshi Y., Minoda H., Arai Y. and Nagayama K., *Micron* **43**,(2012) 1092-1098



**Figure 1.** A comparison of the surface of the phase plates after annealing at 350°C(a) and 700°C(b). 5nm of Ga was deposited before annealing.



**Figure 2.** A comparison of the FFT patterns of the TEM images of the amorphous film (a) without and the (b) with a phase plate.