doi:10.1017/S1431927611006921
Microsc. Microanal. 17 (Suppl 2), 2011

New Type of Silicon Based Zernike Thin Film Phase Plate

S.H. Irsen*, S. Pattai**, P. Kurth**, J. Wamser**

*Electron Mircroscopy and Analytics Lab, Research Center Caesar, 53175 Bonn
**KonTEM Phase Contrast Systems, 53175 Bonn

Weak amplitude and phase objects like unstained vitrified biological samples suffer from poor object contrast when examined with conventional transmission electron microscopy (TEM). Contrast can be enhanced by introducing a phase plate into the back focal plane of the TEM [1], which adds an additional phase shift of pi/2 to the scattered electrons and thus greatly enhances image contrast. The phase shift can either be applied using an electrostatic micro lens (Boersch-type) or a thin holey film (Zernike-type) [2]. In Zernike-type phase plates, the phase shift of the scattered electrons is caused by the electrostatic potential of the phase plate material. Amorphous carbon films are prominent materials for fabrication of Zernike phase plates. Contamination and ageing, however, are known problems of these thin films and limit their widespread use.

We present a new type of a Zernike thin film phase plate based on amorphous silicon instead of carbon. We used a 5 nm silicon membrane as base material. To enhance electrical conductivity and to fine-tune the desired phase shift, membranes were coated with one or two layers of amorphous metals [3]. Various coating materials were tested for their suitability. Chromium turned out to be the best choice. Layers as thin as 2 nm showed sufficient homogeneity and electrical conductivity. Layers between 2 and 10 nm were fabricated using a standard sputtering device (Baltec MED010). Optimized pi/2 phase shift of the silicon-metal film was achieved at a chromium layer thickness of 8 nm.

Finally, holes of a diameter of 300 - 1200 nm for the unscattered electron beam were milled into the film using a focused ion beam workstation. The energy of the gallium ions was set to 5 kV to avoid additional thickening of the hole edges.

The main advantage of using silicon as phase plate material is the improved long-term stability of the films. In our first tests, we used a single film over a period of several weeks without visible contamination or significant damage of the hole edge. Additionally, the silicon films are stable against cleaning in Ar/O plasma. Selected phase plates were cleaned using a standard plasma cleaner (Fischione 1020) using process times up to 10 min without destroying the film.

Fig. 1 shows first application results of the new phase plate. Plastic sections of mouse retina were used to show the contrast improvement achieved by using the phase plate. Contrast improvement was calculated using profile plots at selected areas of the images. A region with membrane features was chosen for the calculation; contrast changes can more easily be seen in showing such details. Contrast was calculated after normalization of the images ($c = (max - min) / (max + min)$). A contrast enhancement of $c = 2.4$ was achieved by using the phase plate.

References

[1] R. Danev, K. Nagayama, *Journal of the Physical Society of Japan* 70 (2001) 696
[2] E. Majorovits et al., *Ultramicroscopy* 107 (2007) 213
[3] R. Danev, K. Nagayama, *Ultramicroscopy,* 88 (2001) 243
[4] This research was supported by the federal ministry of economics and technology (EXIST grant 03EFT4NW32)

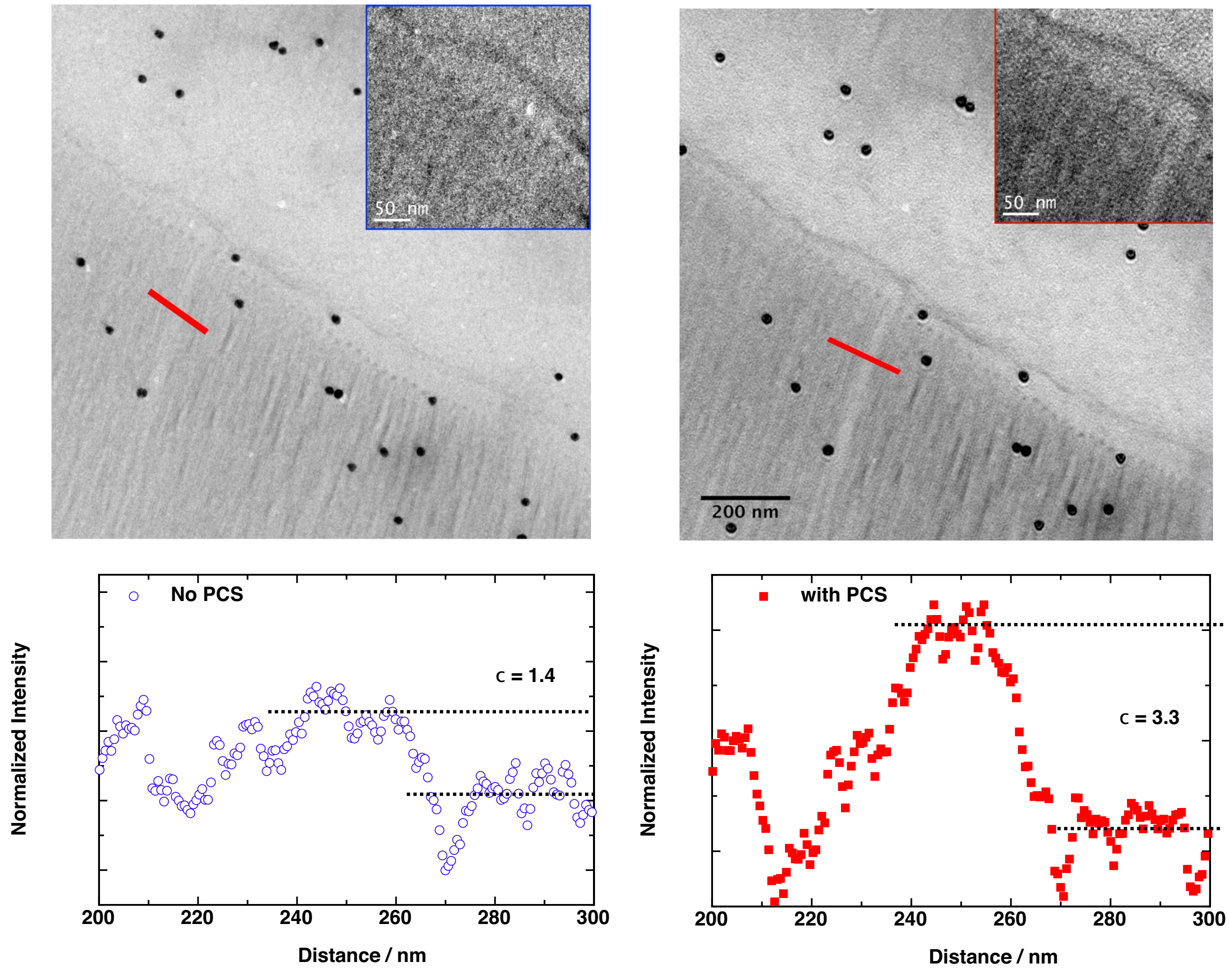

FIG. 1. First application example of new silicon based thin film phase plate. Sample: mouse retina, RIM region; sample was high pressure frozen and freeze substituted. Section thickness = 200 nm. Left image shows part of an outer segment rod of mouse retina using normal bright field TEM while the right image shows the same region using the new phase plate. The insets show a magnified detail of the image. The two graphs show line profiles at the region marked with red lines.

https://doi.org/10.1017/S1431927611006921 Published online by Cambridge University Press