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Thin-Film-Based Phase Plates for Transmission Electron Microscopy Fabricated From Metallic Glasses

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Intense development of physical phase plates (PPs) in the past decade led to substantial improvements in transmission electron microscopy (TEM) imaging of weak-phase objects. Research has focused on thin-film PPs, which are typically fabricated from amorphous carbon (aC)-films [1]. Amorphous carbon has two important properties, which are essential for phase-contrast TEM: A sufficiently high electrical conductivity and an amorphous structure to avoid Bragg diffraction in the PP material. Thin-film PPs based on aC-films have already become widely accepted to enhance the contrast of weak-phase objects in TEM [2]. However, the irradiation with high-energy electrons initiates a steady, irreversible degeneration of the aC-film, which reduces the lifetime of aC-film-based PPs. Therefore, recent investigations have focused on the search of alternative materials with an improved material stability [3,4].

This study, for the first time, presents thin-film PPs fabricated from a metallic glass alloy. Metallic glasses are characterized by a high electrical conductivity and an amorphous structure. Moreover, structural degradation under the intense electron-beam is not expected if the crystallization temperature is high enough. $Zr_{65.0}Al_{7.5}Cu_{27.5}$ (ZAC) was chosen for its favorable properties and its high crystallization temperature of 437 °C [5]. We have applied Hilbert PPs (HPPs) in this work, which consist of a micro-structured thin film located in the back focal plane of the objective lens [6]. The film thickness is adjusted in such a way, that a phase shift of π is imposed on the electrons in one half of the diffraction pattern except for the zero-order beam. This yields an overall phase shift of π/2 for spatial frequencies above the cut-on frequency.

The ZAC-film was sputtered on a cleaved mica-substrate and floated on a Cu-grid. Using a focused ion-beam system, rectangular windows were structured into the ZAC-film, which yields HPPs in several meshes of the Cu-grid. The Cu-grid was mounted in an objective aperture stripe and implemented in the back focal plane of a *Philips CM200 FEG/ST*. At an acceleration voltage of 200 kV, the ZAC-film of 24 nm thickness induces a phase shift close to π.

Fig. 1a shows a cross-section TEM image of a ZAC-film sputtered on a Si-substrate. Even short periods at ambient air lead to an oxide layer of 4 nm thickness, which appears with intermediate gray contrast in Fig. 1a. The oxygen is also visible in the composition profile shown in Fig. 1b, which was obtained by energy dispersive X-ray spectroscopy (EDXS). The electrically insulating oxide layer causes electrostatic charging of the ZAC-film, which affects its phase shifting behavior. Therefore, a thin aC-coating was applied to the ZAC-film. Fig. 2a depicts the power spectrum of an amorphous test object, which demonstrates the desired phase shifting properties. The power spectrum is subdivided in a central stripe (red) and outer areas (green) with Thon-rings shifted by π/2. The complementary behavior in the two regions is also demonstrated by the azimuthally averaged intensity profiles shown in Fig. 2b.

Although amorphous carbon was not fully removed from the PP-production process, the properties of metallic glasses are promising to improve the applicability of thin-film PPs for phase-contrast TEM [7].

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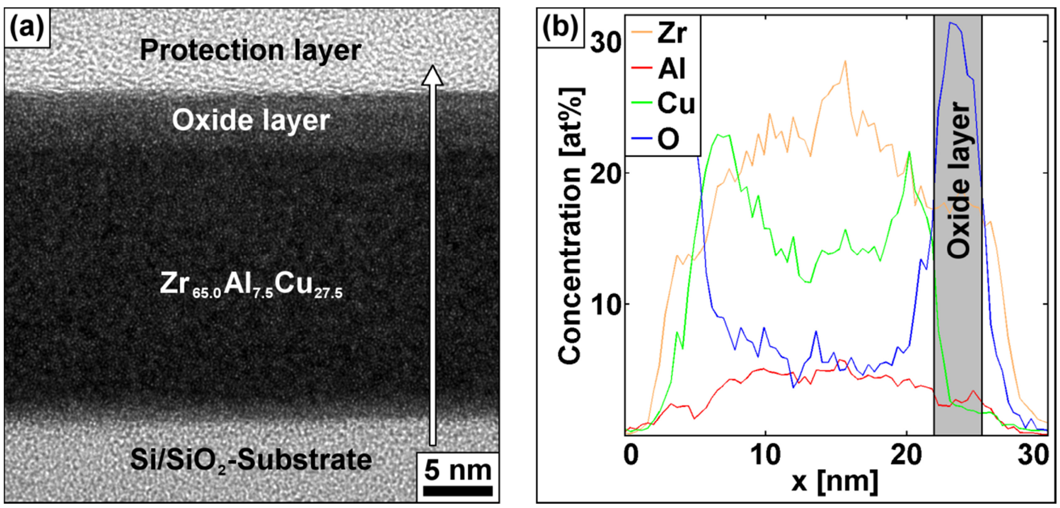

Figure 1. Formation of oxide layers at the surface of ZAC-films. (a) Cross-section TEM image of an oxidized ZAC-film with a Pt/C-protection layer on top. (b) Composition profile along the white arrow in (a) obtained by EDXS measurements.

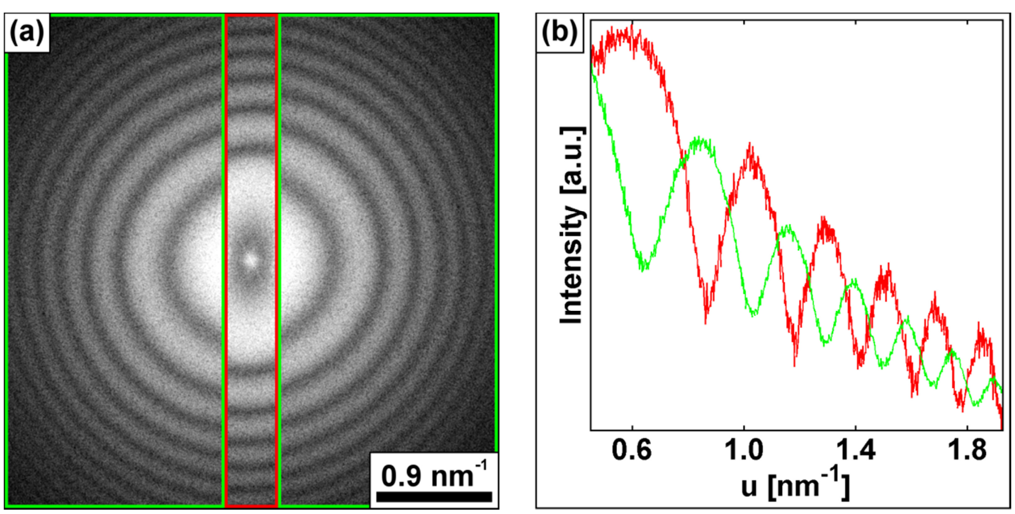

Figure 2. Phase shifting behavior of the aC-coated ZAC-film-based HPP. (a) Power spectrum of a phase-contrast TEM image of an amorphous test object. (b) Azimuthally averaged intensity profiles taken from the Thon-rings in the red and green regions of (a).