

Fabrication and characterization of an adaptive thin-film electrostatic phase plate

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In this work, we establish a robust protocol for designing and fabricating adaptive electrostatic thin-film phase plates, as demonstrated by the first experimental realization of such a device and its characterization. A phase plate placed before the sample in a transmission electron microscope (TEM) can shape the beam to create user-configured irradiation with predesigned amplitude and phase distribution. It is, therefore, possible to use a phase plate to compensate for aberrations of the beam, which are a common problem in electron microscopy. The electron-optical index of refraction is proportional to the electric and magnetic potentials. Thus, the active part of a phase plate is designed to generate a local electric and/or magnetic field. By using a specific set of electrodes, the imprinted phase distribution of the electron beam can be adjusted externally by changing the potential applied to the electrodes. This is denoted as an adaptive electrostatic phase plate. Here, we present the design, fabrication, and characterization of an adaptive thin-film electrostatic phase plate, with gold electrodes supported on a thin silicon nitride membrane. Our design consists of six electrodes positioned as concentric and equidistant circles. We describe the fabrication of the phase plate, with particular focus on the less conventional steps involved. After careful process optimization, the fabricated phase plate features largely match the design, and approximately 95% of the 174 chips fabricated on each wafer show no visible defects in the active area. We characterize the functionality of the fabricated phase plate using both computational (COMSOL Multiphysics) and experimental (Fresnel imaging) methods. The phase profile reconstruction using the transport of intensity equation method generally follows the predicted phase profile. The desired quartic phase profile is partly achieved, although the presence of electrodes increase noise, and the electrode contacts introduce asymmetry. This opens up opportunities for further optimization before implementing the phase plates in a TEM for spherical aberration correction.

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I. INTRODUCTION

The advent of electron phase plates revolutionized electron microscopy by providing the ability to introduce a controlled local phase shift to the electron wave function [1]. Phase plates provide us with new imaging and analysis modes [2], using components and methods comparable with their optical counterparts. Shaping light beams has been conceptualized in terms of *adaptive optics*, where a feedback loop drives real-time beam optimization for, e.g., focal spot detection and position control. A focused monochromatic beam can be reshaped into various

configurations with controlled phase and amplitude. This has been achieved, for example, using diffractive optical elements [3], spatial light modulators [4], and, more recently, metasurfaces [5]. Similarly, phase plates are used to shape the electron beam into user-prescribed configurations with defined phase and amplitude distributions. In this case, the electrons are modulated in relation to the electron-optical index of refraction, which is proportional to the electric and magnetic potentials [6].

By installing the phase plate before the sample, the electron beam can be shaped to produce a predefined irradiation. An example, which is the focus of this manuscript, is to modulate the irradiation to compensate for the detrimental aberrations of the optical system occurring further down the column [7]. A primary application would be to form a corrected subangstrom probe in a scanning transmission electron microscope (STEM) without the need for expensive probe corrector hardware. Other examples include shaped probes in a STEM to match and highlight

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specific atomic-scale features of the sample [8], the irradiation of the sample with Bessel beams, and electron vortices in general [9,10]. In terms of analysis, where the phase plate is installed after the sample, applications range from the contrast enhancement offered by Zernike-type designs [11], to the decomposition of the exit wave into orbital angular momentum (OAM) components provided by the dual-phase-plate system, called an “OAM sorter” [12].

The phase plate functionality originates from local electric and/or magnetic fields that can be established by a specifically designed set of electrodes [13], electrical currents [14], magnetic elements, thickness variations [15–17], or computer-generated holograms as amplitude masks [18]. We differentiate between active and passive phase plates based on their ability to change the electromagnetic field by external control. Passive phase plates shape the electron beam with a predesigned electromagnetic field with fixed intensity and distribution. Active, also known as adaptive, phase plates influence the beam with external electromagnetic field control by intensity and/or shape. According to the frequency of operation, adaptive phase plates can be either static or dynamic.

Adaptive phase plates can be reconfigured in real time by changing the electromagnetic field that they produce. This is a crucial requirement to ensure that a phase plate can be useful in different circumstances, rather than in a single specific situation. The ultimate target is full phase control over the whole electron beam cross section using a large number of “pixels,” i.e., individually addressable areas, which can locally set the electromagnetic field magnitude and orientation [19,20]. However, a phase plate with only a few such pixels is already sufficient for limited real-time electron beam reconfiguration.

The collective area of the pixels, called the active area of the phase plate, should have dimensions matching the size of the electron beam in the chosen conjugated focal plane. The electron beam size when passing through the electron microscope (EM) column ranges from tens of nanometers in the focal plane of the objective lens, up to tens of micrometers in the condenser aperture. The latter is the intended placement of the phase plate discussed in this manuscript. Since the phase plate is placed in the path of the electron beam, it needs to be as transparent to electrons as possible. This is, in fact, one of the primary concerns in all phase plate designs, and probably the most challenging issue to tackle as more flexibility in the beam shaping function requires more pixels, and therefore more electrodes, which, in turn, brings more material into the beam path in the form of leads, contacts, thickness of the support for structural integrity, etc. Ideally, the least invasive phase plate would be a vacuum region with an electric field that is established and controlled by electrodes that are entirely outside the active area. This, however, poses a fundamental limitation in the beam shaping capability of the device, as in vacuum the potential

satisfies Laplace’s equation, implying that it can only produce a harmonic modulation of the phase that lacks the necessary extrema (minima or maxima) to compensate for circularly symmetric aberrations.

Thus, the electrodes need to be placed either around an opening that the beam passes through or over an ultrathin support. These prerequisites for configurability come with challenges in terms of the design, fabrication, and actuation of the phase plate, more specifically in terms of the layout and deposition of conducting micro- or nanoscale patches that allow for individual voltage gating while avoiding crosstalk and parasitic networks.

Beam shaping using electrodes was recently reported by Tavabi *et al.* [9]. The active phase plate area consisted of a pair of suspended “chopsticks” electrodes protruding over a hole in the silicon chip, complemented by a few electrodes disposed around the hole. This phase plate design was employed for generating a vortex beam.

An interesting design for spherical aberration correction was published by Ribet *et al.* [7]. The proposed device consisted of a series of two-terminal electrostatic phase plate devices arranged into a larger phase plate. The device was meant to be inserted into the probe-forming aperture of a STEM. According to simulations, the phase plate device could successfully correct for spherical aberrations. Similarly, Ibáñez *et al.* proposed the use of programmable phase plates consisting of arrays of electrostatic phase-shifting elements for TEM aberration correction [21]. Numerical analysis shows that such designs can flatten the electron wave front up to relatively high opening angles and can provide probe sizes of less than 1 Å. Another interesting recent example of phase plates for electron beam shaping comes from Ruffato *et al.*, who explored designs with stacked phase plates for complex beam shaping [22]. Despite being very promising, based on theoretical results, none of these examples were implemented in practice, most likely due to fabrication challenges.

Commercially available silicon nitride (SiN_x) TEM membranes have a thickness of around 50 nm. However, thinner membranes are known to yield a better contrast in TEM imaging due to reduced scattering [23–25], but they show reduced membrane stability. Cleanroom technologies have been used for the fabrication of TEM chips, including both membranes and electrodes [26]. Yang *et al.* [27] fabricated one such chip for liquid-cell *in situ* electron microscopy of electrochemical processes, where the membrane consisted of a SiN_x thin film, and gold electrodes were present elsewhere on the chip.

In this manuscript, we present the design, fabrication, and characterization of an adaptive thin-film electrostatic phase plate for spherical aberration correction. The phase plate should be placed before the sample and employed as an electron beam shaper for spherical aberration correction of the probe-forming optical system in a STEM.

We describe the fabrication of the phase plate, focusing on challenges and found solutions. We present the theoretical considerations for designing, fabricating, and actuating such a device. We assess the performance of the fabricated phase plate both theoretically and experimentally using COMSOL simulations and electron Fresnel imaging, respectively. The phase shift profile from experimental data matches well with our simulations. However, an undesired breakage of circular symmetry is introduced in the phase plate profile owing to the positioning of our electrode contacts. Thus, a redesign of the phase active area of the phase plate is required before it can be used for its intended purpose.

To the best of our knowledge, this is the first experimental demonstration of an adaptive thin-film electrostatic phase plate. The developed fabrication protocol is extremely robust, allowing for the fabrication of 174 phase plate chips on each 100-mm wafer employed, out of which approximately 95% display no visible defects in the active area. The established fabrication protocol and experimental limitations identified provide a framework that should allow for improved functionality through, e.g., shape and topology optimization of the phase plate using gradient-based optimization and the inverse design approach.

II. DESIGN AND FABRICATION

A pattern consisting of concentric circles with equidistant spacing was chosen as the starting point for spherical aberration correction because of the radial symmetry of the electron beam. The overall chip size ($4.00 \times 4.65 \text{ mm}^2$) and choice of six electrodes were made to interface with a Protochips Fusion Select TEM holder [Fig. 1(a)]. The electrodes were connected to the Protochip Fusion Select holder through a pin system with six contacts. These were placed in specific locations, which were extrapolated from a Fusion Select E-chip (see SI1 in the Supplemental Material [28]). To minimize the risk of disconnections in the electrode contacts, all contact pads were kept as large as possible up to the active area of the phase plate.

The design of the active area of the phase plate [Fig. 1(b)] was made taking into account the electron beam size in the condenser aperture, which is the intended position during operation. The thin-film membrane dimension was set to $100 \times 100 \text{ }\mu\text{m}^2$ to ensure that the electron beam passed through only the membrane and did not interact with the silicon part of the chip. A critical consideration was how to connect the circular electrodes to an external voltage source while preserving the circular symmetry as much as possible. Owing to geometrical limitations and to keep the electrode shape as close as possible to a circle, we opted to implement the contact leads for the outer ground electrode and for the five inner electrodes on opposite sides of the chip. The large outermost ring electrode, which is used for ground connection and denoted as “GND,” has

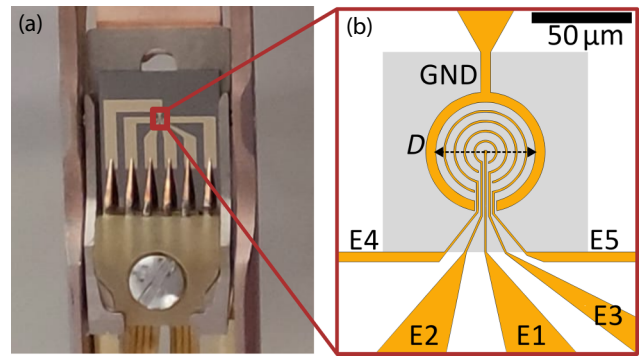


FIG. 1. (a) The thin-film adaptive electrostatic phase plate installed in the Protochip Fusion Select transmission electron microscope holder, with the different electrode pads connected to separate pins for individual potential control. (b) Schematic of the electrodes in the active area of the phase plate, with the working electrodes (E1–E5) and the grounded electrode (GND). The gray square indicates the placement and size of the membrane and D is the inner diameter of the GND electrode.

a width of $5 \text{ }\mu\text{m}$ and an inner diameter of $50 \text{ }\mu\text{m}$. Inside the active area, there are five electrodes in addition to the GND: four rings and a center disk. The rings each have a width of $1 \text{ }\mu\text{m}$, the center disk is $2 \text{ }\mu\text{m}$ in diameter, and the gap between two adjacent electrodes is $4 \text{ }\mu\text{m}$. These electrodes are denoted as E1 to E5, with the center disk being E1. The electrode width was chosen based on the resolution limit of the maskless aligner (MLA) system employed for fabrication.

Based on the design and several experimental considerations, we developed a process flow for the fabrication of a thin-film adaptive electrostatic phase plate, which is shown in Fig. 2 and detailed in the following. High-quality silicon wafers, $100 \pm 0.3 \text{ mm}$ in diameter, double-side polished (DSP), $\langle 100 \rangle$ -oriented, $350 \pm 15 \text{ }\mu\text{m}$ thick, phosphorous doped (n -type), $1\text{--}20 \text{ }\Omega \text{ cm}$ resistivity, from Sievert Wafers, were used as the substrates [Fig. 2(a)].

SiN_x was deposited on both sides of the wafer by low-pressure chemical vapor deposition (LPCVD) (Tempress LPCVD nitride furnace). The deposition was performed as a batch process with 25 wafers at $830 \pm 20 \text{ }^\circ\text{C}$, 200 sccm flow of dichlorosilane, 50 sccm flow of ammonia, at 150 mTorr pressure [Fig. 2(b)]. To reduce membrane-induced scattering as much as possible while maintaining membrane integrity, we deposited around 30 nm of SiN_x , which resulted in a membrane thickness of about 25 nm of SiN_x after etching (see SI2 in the Supplemental Material [28]). The SiN_x on the back side was used as etching mask, while the front side became the thin-film membrane supporting the active area of the phase plate.

The back side opening of the SiN_x membrane was created through photolithography and dry etching [Fig. 2(c)]. First, the wafer was pretreated with hexamethyldisilazane (HMDS). For this, the wafer was first baked at $120 \text{ }^\circ\text{C}$,

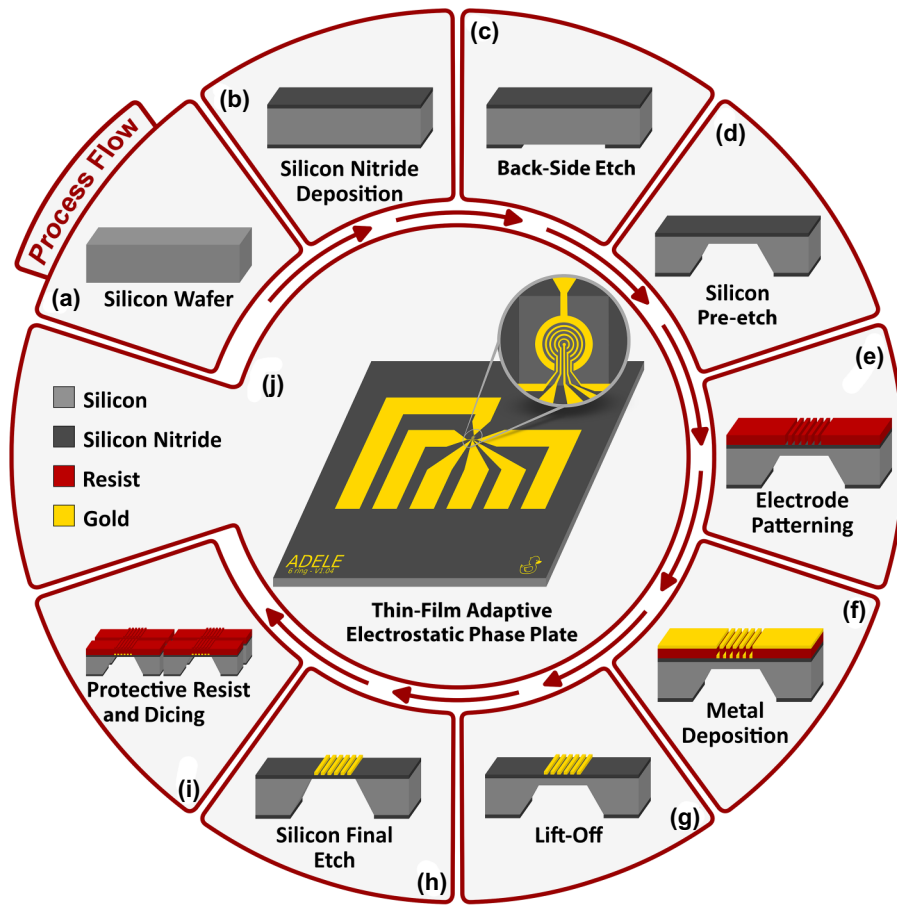


FIG. 2. Schematic of the process flow for the fabrication of a thin-film adaptive electrostatic phase plate from a silicon wafer to a diced chip. The colors represent different materials as seen on the left. (a) The process starts with a double-side polished silicon wafer. (b) A layer of SiN_x is deposited on both sides of the wafer using low-pressure chemical vapor deposition. (c) The nitride on the back side of the wafer is opened through dry etching and used as a mask for KOH etching. (d) Silicon etching using KOH for approximately 90% of the wafer thickness. (e) The nitride on the front side of the wafer is to be used as the membrane, on which an electrode is patterned in resist using image reversal. (f) The front side is covered in metal using e -beam evaporation. (g) Lift-off is used to remove the resist and extra metal on the front side. (h) Freeing of the front side membranes by KOH. The front side is protected in a special holder during this etch. (i) Dicing of the wafer into $4.0 \times 4.65 \text{ mm}^2$ chips. The wafers are coated with a thick layer of resist to improve stability during dicing. (j) Final phase plate design including an inset with a magnified view of the electrodes on the membrane.

0.35 bar. Then 10000 sccm N_2 was bubbled through liquid HMDS before injection into the chamber and the reaction lasted until ambient pressure was reached. The chamber was then purged using clean N_2 gas. Finally, the wafer was cooled on the 20°C cool plate for 20 s. Next, 3 ml of AZ MiR 701 resist was dispensed onto the wafer by spin coating (Süss MicroTec, model Gamma 2M) at 800 rpm, followed by an acceleration at 2000 rpm/s to 4600 rpm for 30 s. The wafer was moved onto a hot plate for 60 s at 90°C for soft baking. Finally, the wafer was cooled on the 20°C cool plate for 20 s. The resulting resist height was approximately $1.5 \mu\text{m}$. The exposure mask pattern was designed in the CLEWIN software and exported to the gds format. A square pattern with a size of $590 \times 590 \mu\text{m}^2$ was selected to produce $100 \times 100 \mu\text{m}^2$ membranes after wet etching. The AZ MiR 701 resist was exposed in the desired

pattern by direct writing using a 405-nm light source (Heidelberg Instruments MLA150 Maskless Aligner, s/n HI 1155). The wafer was aligned to the major flat and exposed at a dose of $175 \text{ mJ}/\text{cm}^2$ at 0 defocus. A postexposure bake was performed at 110°C for 90 s, after which cooling was performed on the 20°C cool plate for 20 s. The pattern was developed using puddle development. AZ 726 MIF was dispensed at 225 ml/min for 3 s while the wafer rotated at 30 rpm, forming a puddle on the wafer. The wafer was developed for 60 s with a 2 s, 30 rpm rotation after 30 s. The developer was removed by a 3 s spin-off at 4000 rpm, after which the wafer was spun at 4000 rpm for 30 s while dispensing 500 ml/min deionized (DI) water. The wafer was then slowed to 3000 rpm, where it was dried for 10 s using a 50 l/min flow of N_2 . The thin-film nitride was then etched using the AZ MiR 701 pattern as a mask

in a deep reactive-ion etching system (STS MESC Multiplex ICP). This dry-etch step was done for 35 s with 700 W coil power, 100 W platen power, 0 °C platen temperature, 4 mTorr pressure, 5 sccm C₄F₈ flow, 174 sccm He flow, and 4 sccm H₂ flow. The resist mask was then removed by plasma ashing (TePla model 300 Semi Auto Plasma Processor) for 30 min at 1000 W power, 600–750 mTorr pressure, 400 sccm O₂ flow, 70 sccm N₂ flow.

The SiN_x membrane was fabricated through wet etching of the silicon using the back side silicon nitride pattern as the etching mask. Normally, an immediate full etch could be used for opening the membranes [29]. However, etching all the silicon at this stage would prove problematic for subsequent lithography steps. First, having freestanding membranes removes the ability to use an automatic spin coater and developer for subsequent steps. Second, applying a vacuum to the back side of the wafer during subsequent steps would create a curvature in the membrane, impacting the lithography pattern and potentially resulting in membrane breakage. Alternatively, the wet etch could be done as the last step in the process. However, protection of the patterned side is needed for etching after the fabrication of the electrodes, since contact with KOH would damage the metal layer [30] (see SI3 in the Supplemental Material [28]). To minimize the challenges posed by both approaches, we chose to do a pre-etch step where approximately 90% of the silicon was removed, followed by a final etch and membrane release after the electrode patterning step. For the first etch step [Fig. 2(d)], the wafer was lowered into a wet-bench bath with 28% KOH at 80 °C for 4 h. The wafer was then cleaned using a deionized water bath, where it was submerged for 300 s, then placed in a spin dryer, where the wafer was first rinsed with deionized water while rotating at 500 rpm, followed by 180 s of spin drying at 1800 rpm. The etch depth was checked with a Dektak XTA stylus profiler and recorded. The target etch depth was around 312 μm. To remove residual ions from the wet-etch step, the wafer was cleaned using a wet bench containing sulfuric acid at 80 °C for 10 min. Just before the wafer was submerged, ammonium persulfate was added to the bath. The wafer was cleaned using a deionized water bath, in which it was submerged for 300 s. The wafer was then placed in a spin dryer, where it was first rinsed with deionized water while rotating at 500 rpm, then spin-dried for 180 s at 1800 rpm.

For the electrode patterning step [Fig. 2(e)], the wafer was first pretreated with HMDS, as described above. Then, 3 ml of AZ 5214E resist was deposited on the front side of the wafer by spin coating (Süss MicroTec, model Gamma 2M) at 800 rpm, followed by an acceleration at 2000 rpm/s to 4500 rpm for 30 s. The wafer was moved onto a hot plate for 60 s at 90 °C for soft baking. Finally, the wafer was cooled on the 20 °C cool plate for 20 s. The resulting resist height was around 1.5 μm. The AZ 5214E resist was then exposed in the desired pattern (see SI4 in

the Supplemental Material [28]) by direct writing using a 405-nm light source (Heidelberg Instruments MLA150 Maskless Aligner, s/n HI 1155). The wafer was aligned to the major flat and exposed at a dose of 50 mJ/cm² at 0 defocus. Finally, the pattern was reversed from a positive to a negative pattern by a reversal bake (Süss MicroTec Gamma 2M developer), followed by a flood exposure (Süss MicroTec MA6/BA6 aligner). The reversal bake was performed at 110 °C for 90 s, after which cooling was performed on the 20 °C cool plate for 20 s. The wafer was then flood exposed for 49 s with a 500-W Hg-Xe lamp with 365-nm wavelength and 11-mW/cm² intensity. Development of the structures was done as described for the previous lithography step. We chose to use image reversal for electrode patterning because we found it to be the best approach for obtaining a negative sidewall, which is critical to ensure good lift-off.

The metal for the electrodes and contacts was deposited using *e*-beam evaporation (Ferrotec, Temescal FC-2000 *e*-beam evaporator) [Fig. 2(f)]. A 5-nm adhesion layer of chromium was deposited at a rate of 2 Å/s, circular sweep, while the wafer was rotating at 15 rpm. A 25-nm gold layer was then deposited at a rate of 2 Å/s, star sweep, while the wafer was rotating at 15 rpm (see SI5 in the Supplemental Material [28]). We selected gold as the electrode material due to its excellent electrical properties and low reactivity, which prevents undesired oxidation during storage. The thickness of the electrodes was chosen to match the thickness of the membrane. While the chromium layer is not necessary for the electrical properties of the electrodes, it improves the adhesion of the gold layer [30], which might otherwise detach during the KOH etch [23].

The gold on top of the resist and the resist itself was removed through a lift-off process [Fig. 2(g)], where the wafer was lowered into a wet-bench chemical bath of Remover 1165 for 25 min. Ultrasound was included during the lift-off to speed up the process. The wafer was moved to an isopropanol chemical bath for 3 min with sonication. Finally, the wafer was cleaned with DI water in a wet bench for 300 s. The wafer was placed in a spin dryer, where the wafer was first rinsed with DI water while rotating at 500 rpm, then spin-dried for 180 s at 1800 rpm.

As previously mentioned, the last fabrication step was a wet-etch step done to release the SiN_x membrane. For this purpose, we used a polyether ether ketone protective holder to cover the front side of the wafer and prevent direct contact between the metal electrodes and the KOH solution (see SI6 in the Supplemental Material [28]). However, the protective holder was not fully leakproof, so the etch time for this step should be reduced. Additionally, the protective holder only allowed for the processing of two wafers at a time, whereas standard silicon wet etching allows for batch processing (up to 25 wafers). This is why we chose to do the wet etching in two steps at different stages of the process, a pre-etch step where approximately

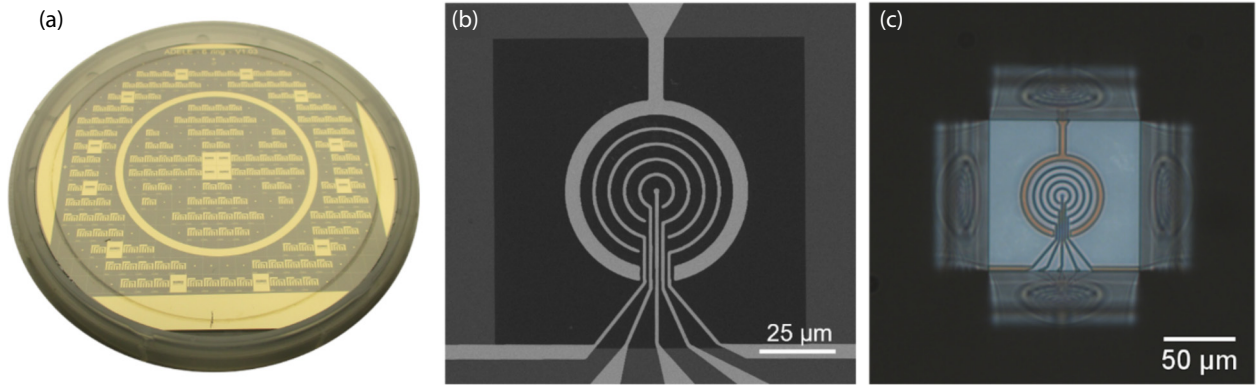


FIG. 3. (a) Photograph of the full 100 mm wafer before dicing. (b) Scanning electron microscopy image of the front side of a phase plate chip after dicing and removal of the protective resist. The dark square is the SiN_x membrane and the light gray patterns are the metal electrodes. (c) Optical microscope image taken from the back side of the wafer. The electrodes are clearly visible through the membrane, indicating full removal of the silicon in this area.

90% of the silicon was removed, [Fig. 2(d)], followed by a final etch and membrane release after the electrode patterning step [Fig. 2(h)]. The wafer placed in the protective holder was lowered into a chemical wet bench containing 28% KOH at 80 °C. The wafer was removed after approximately 40–50 min, once the hydrogen bubbling from the etching ended. The exact etch time needed depended on the residual wafer thickness. The wafer was cleaned by lowering it into a deionized water bath for 300 s twice, first in the protective holder, then by itself. The wafer was then placed in a spin dryer, where it was first rinsed with deionized water while rotating at 500 rpm, then spin-dried for 180 s at 1800 rpm. To remove residual ions from the wet-etch step, the wafer was immersed in a 7.4% hydrochloric acid solution for 10 min, and then submerged in a deionized water bath for 300 s and spin-dried for 180 s at 1800 rpm.

Before dicing, a protective layer of resist was spun onto the front side of the wafer, then 5 ml of AZ 5214E was manually spin coated onto the wafer (SÜSS Microtec Lab-Spin6). The wafer was accelerated to 500 rpm and spun for 60 s. The wafer was then soft baked at 90 °C for 60 s, after which wafer was cooled on the 20 °C cool plate for 20 s. This resulted in a final resist thickness of approximately 4 μm. This resist layer was meant to reinforce and stabilize the membranes during dicing. Additionally, the resist functioned as a protective layer against contamination during storage. The wafer was diced into chips using the DISCO Automatic Dicing Saw, DAD 3241 [Fig. 2(i)]. For this, the wafer was installed on UV-releasable foils on steel rings by carefully sticking the wafer to the foil and avoiding making air bubbles. A 27HEEF blade was used for cutting at a speed of 30 mm/s. To release the tape, the wafer was run through a Powatec UV Curing System U200 for 10 s. Based on optical microscopy inspection, approximately 95% of the membranes (165 out of 174 chips) were intact after dicing wafers covered with this protective coating. An attempt at dicing the

wafer without the protective coating resulted in broken membranes throughout the wafer. Before TEM characterization, the protective resist was removed by subsequent immersion in acetone (15 min), isopropanol (5 min), and deionized water (5 min). Finally, a 5-min argon plasma cleaning was done.

Figure 3 shows images of the fabricated phase plate chips: Fig. 3(a) shows a photograph of a full 100 mm wafer after the final silicon etch step, Fig. 3(b) shows the active area from the front, and Fig. 3(c) shows the active area as seen from the back. The wafer contained 174 phase plate chips and 16 test pattern chips, which appear golden. In total, 190 chips were arranged in a grid-like pattern for dicing on the mechanical saw. The large golden ring is where the tool vacuum chucks were, and no chips were placed around this area.

III. THEORETICAL AND EXPERIMENTAL CHARACTERIZATION

Phase plates are used to modulate the phase shift of the electron wave function that either illuminates or emerges from a sample. Here, we aim to introduce a controlled negative spherical aberration to the irradiation that compensates for the positive aberration of the imaging system. Since the transfer function $T(q)$ of a lens affected by spherical aberration is [31]

$$T(q) = e^{i\frac{\pi}{2}C_S\lambda^3q^4}, \quad (1)$$

where C_S is the spherical aberration coefficient, λ is the electron wavelength, $q = \theta/\lambda$ is the spatial frequency, and θ is the scattering angle, our goal is to engineer the phase plate to produce a phase shift that decreases with the fourth power of distance from the center. The distance is related geometrically with spatial frequency through the relation $r = \lambda f q$, where f is the camera length of the

electron-optical setup. The condenser aperture plane and the sample plane are conjugate object planes. Therefore, the (de)magnification factor of the condenser system controls the spatial extent of the irradiation patch. The magnitude of the phase difference between the optic axis and the boundary of the irradiation, which coincides with the outer diameter of the device (GND electrode), is set by the spherical aberration coefficient that we aim to correct for a given electron wavelength.

The first concern was whether the desired phase profile could be achieved using this limited number of discrete electrodes. In practice, denoting the radius of the phase plate active area as R (coinciding with the inner radius of the GND electrode), we want the beam to acquire the following phase profile:

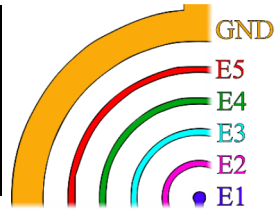
$$\varphi(r) = \varphi_0 \left[1 - \left(\frac{r}{R} \right)^4 \right]. \quad (2)$$

To achieve this, we had to bias each electrode independently, with a particular voltage configuration that produced the target phase shift profile. Since the relationship between phase and potential involves the whole space surrounding the device and is nonlinear, our task was not a simple matter of tuning the biases with a fourth-power dependence on electrode radius. Thus, the potential set to phase shift relationship had to be explored numerically to identify if it were possible to find an optimized set of bias potentials that produced a good approximation of a quartic function as a phase shift.

The optimization of the applied potential on the electrodes was conducted using advanced machine learning techniques within the TensorFlow library in PYTHON. A hybrid machine learning approach was employed, featuring a convolutional neural network (CNN) as its core component. This CNN, trained on over 10^5 two-dimensional simulations, used inputs of random bias voltages applied to six electrodes to predict the resulting total phase shift. The network architecture comprised five layers, including average pooling and Leaky ReLU activation functions, which enabled it to effectively map the complex relationships between phase shifts and electrode biases. The initial predictions provided a solid baseline from which to start a gradient-based optimization process in COMSOL. COMSOL optimization involved adjusting parameters to correct any inaccuracies from the initial machine learning predictions, ultimately refining the outcomes. The optimized potential values required to achieve a quartic phase shift for the design employed in this work are shown in Table I. The main advantage of using a CNN instead of simply selecting a random set of starting potentials for gradient-based optimization is the computational time. Gradient-based optimization requires a rerun of the entire optimization with a new starting set of values if a different phase profile is desired. Instead, the CNN can output

TABLE I. Applied voltages on E1–E5 and GND electrodes. The same values were used for both simulations and experimental data acquisition.

Electrode	Applied voltage (V)
GND	0.0
E5	7.6
E4	8.0
E3	7.2
E2	6.4
E1	6.0



reasonable voltages for obtaining any target phase profile almost instantly. This makes it possible to quickly evaluate if a phase plate could be used to generate other types of phase profiles, and what voltages this would require.

Figures 4 depicts the phase shift obtained from simulations using optimized bias potentials and the concentric ring electrode design. For a more accurate representation of the phase, the wrapped phase shift (cosine of the corresponding phase) is depicted in Fig. 4(f). As shown in Fig. 4, the desired continuous quartic phase shift profile can be approximated well using the concentric ring design, despite the discrete nature of the potential applied and the limited number of electrodes used. In this simplified design, each ring is treated as an electrode with a fixed Dirichlet boundary condition (specified potential). This idealized configuration is not feasible experimentally, because in a real device each ring must be connected to external contacts.

Simulation results for the fabricated design, which includes external electrode connections, is presented in Fig. 5. The designed electrode layout was imported into COMSOL. The geometry was positioned within a $2 \times 2 \times 2$ -mm³ cubic domain that mimics the microscope's vacuum environment. The boundary conditions were specifically tailored to the EM setup: the underside of the device chip, where it contacts the TEM holder, was considered grounded; the device's electrodes were set to defined potentials; and the boundaries representing the vacuum domain of the EM chamber were also grounded. To simplify the solver's complexity and effectively manage the boundary conditions for the outer vacuum domain, the entire model was encapsulated within an infinite element domain of size 20 mm expanded in all directions. Since the most critical parts of our simulation are the trajectory path of the electrons and the domain near the device, high precision in simulating the electric potential distribution is essential in these areas. Therefore, we segmented the vacuum domain into two parts: the electron beams' paraxial region, modeled as a cylinder, and the surrounding domain. This segmentation offers two main

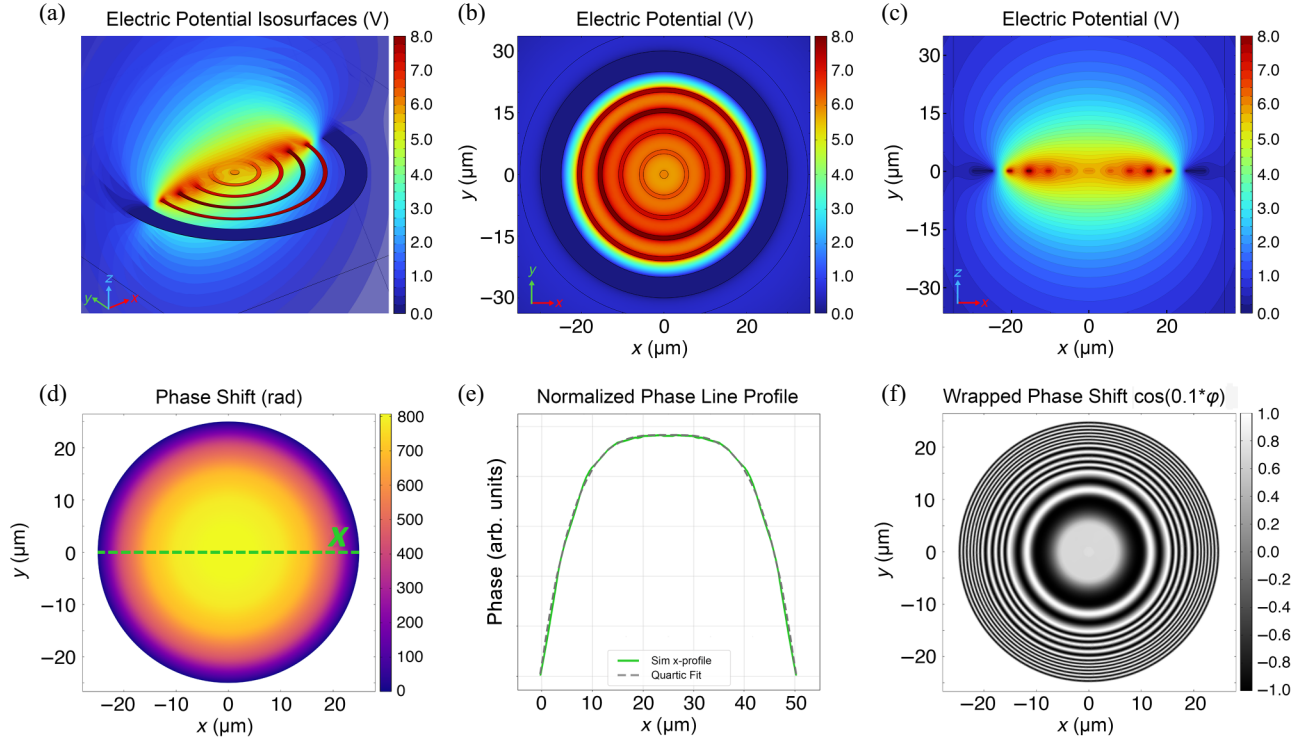


FIG. 4. Simulation results of a simple design with six concentric and equidistant circles for the applied potential values noted in Table I. (a) Electric potential isosurface across the central area of the electrodes. (b) Electric potential distribution over the electrode area in the horizontal x - y plane. (c) Electric potential distribution across the central electrode area in the vertical x - z plane. (d) Unwrapped phase shift over the electrode area in the horizontal x - y plane. (e) Comparison between the simulated normalized phase profile and the desired quartic function along the central x axis. (f) Wrapped phase shift (cosine of the corresponding phase) over the electrode area in the horizontal x - y plane.

advantages. Firstly, it allowed us to refine the mesh elements with “extremely fine” element size in the paraxial domain, resulting in higher precision of the simulation. Secondly, this cylindrical domain was used for the calculation of the projected potential along the electron trajectory using the general projection feature in COMSOL. The rest of the domain was meshed using the “finer” element size to maintain computational efficiency.

For high-precision modeling, the devices were designed with rings as the active area and included their lead connections, as all parts of the structure contribute to the overall potential distribution and therefore influence the final phase shift. The applied potentials on the individual electrodes are listed in Table I. The voltages applied show a nonmonotonic voltage trend, with a slow increase between E1 and E4, a small decrease from E4 to E5, and a sharp decrease from E5 to GND, produces a monotonic increase in the phase, which is not intuitive and highlights the complexity of inverse design. This behavior is in agreement with predictions from AI-based modeling.

To calculate the total phase shift, we used the commonly employed projected potential approximation,

which assumes a single, integrated interaction across the domain. The total phase shift is given by the expression

$$\varphi(x, y) = C_E \int_{-\infty}^{+\infty} V(x, y, z) dz, \quad (3)$$

where the factor $C_E = 6.53 \text{ rad}/(\text{V } \mu\text{m})$ at a 300-kV accelerating voltage [31]. This phase shift calculation can be performed directly within COMSOL using the general projection function, enabling efficient optimization of simulation parameters without the need for external programming. The COMSOL files are available from the authors upon request.

For experimental characterization, the phase plates were employed as samples in the TEM, where Fresnel imaging was used for phase retrieval. The chip was inserted into a Protochips Fusion Select holder [Fig. 1(a)]. The holder was then connected via coaxial cables to a voltage source with individual voltage output on up to ten channels (high-precision low-noise dc voltage source, Type BS 1-10-10 from Stahl electronics). The output of the channels (Table I) was controlled through LABVIEW using the program provided by Stahl Electronics.

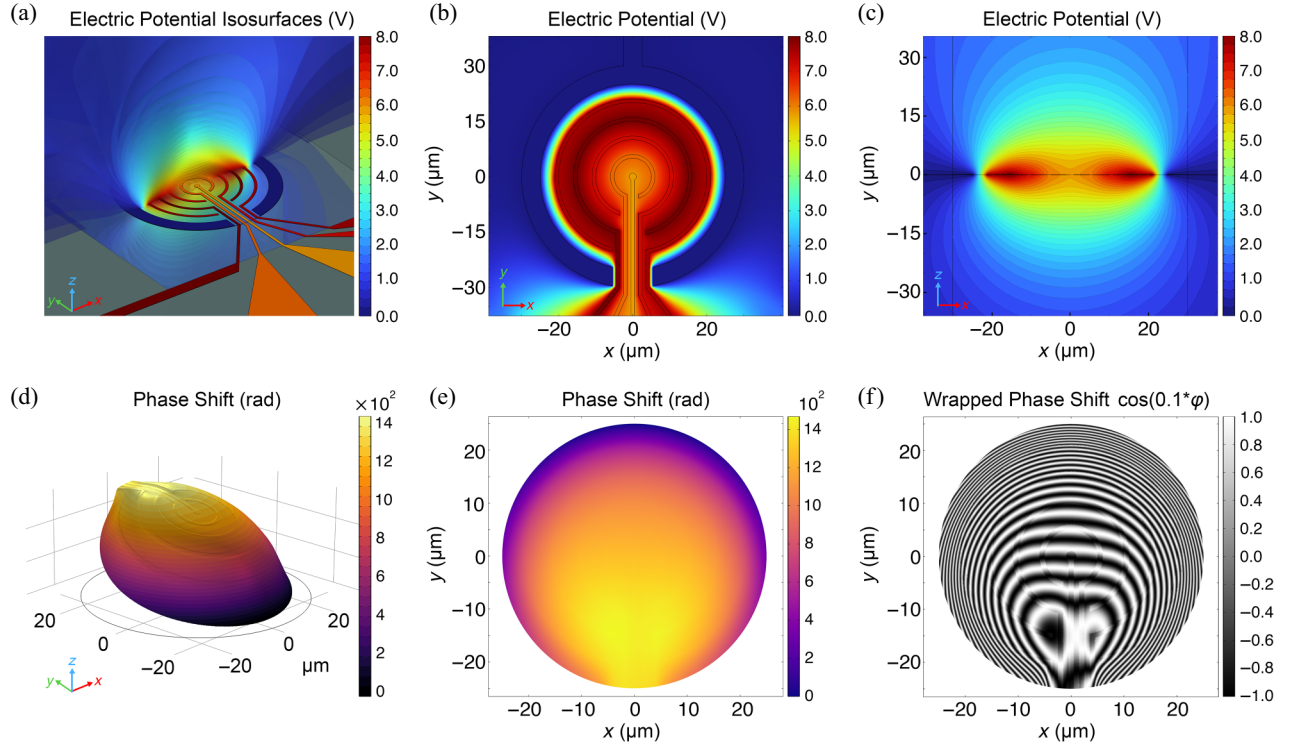


FIG. 5. Simulation results of the phase plate for the applied potential values noted in Table I. (a) Electric potential isosurface across the central area of the electrodes. (b) Electric potential distribution over the electrode area in the horizontal x - y plane. (c) Electric potential distribution across the central electrode area in the vertical x - z plane. (d) Surface plot illustrating the phase modulation of the electron wave front produced by the phase plate. (e) Unwrapped phase shift over the electrode area in the horizontal x - y plane. (f) Wrapped phase shift (cosine of the corresponding phase) over the electrode area in the horizontal x - y plane.

The Fresnel imaging data were acquired by recording a series of TEM images at different defocus values using a FEI Tecnai microscope, equipped with a LaB₆ electron gun, operated at microprobe low magnification (LM mode) and a 200-kV accelerating voltage. Three images, in focus and at 400 μm over or under focus, were captured for positive bias, negative bias, and unbiased configurations. The defocus values were reached by moving the CompuStage up and down without changing the objective lenses, because defocusing by objective lenses at this low magnification introduces noticeable magnification and rotational variation in the images, which hinders the data processing.

The transport of intensity equation (TIE) [32] method for exit-wave reconstruction relies on the acquisition of a pair of slightly defocused images of opposite sign, together with the in-focus image. The purpose of the method is to reconstruct the exit-wave function $\psi = a \exp(i\Phi)$ emerging from the sample, which in this case is the phase plate configured with electrode biases that produce the specific quartic profile necessary for aberration correction. The amplitude is promptly determined by the in-focus image, as its image intensity $I(\mathbf{r}, z=0) = |\psi(\mathbf{r})|^2 = a^2$. The phase, instead, requires a few steps. The TIE equation

reads [32]

$$\begin{aligned} \nabla \cdot [I(\mathbf{r}, 0) \nabla \varphi(\mathbf{r})] &= -\frac{2\pi}{\lambda} \frac{\partial I(\mathbf{r}, z)}{\partial z} \\ &\approx -\frac{\pi}{\lambda Z} [I(\mathbf{r}, Z) - I(\mathbf{r}, -Z)] \\ &\equiv -\frac{\pi}{\lambda Z} \Delta I(\mathbf{r}), \end{aligned} \quad (4)$$

where the axial derivative of the intensity at $z=0$ is approximated by the difference between the over- and underfocus images in the small defocus limit $Z \rightarrow 0$, with Z the chosen defocus distance. The solution of the TIE equation in this approximation is

$$\varphi(\mathbf{r}) = -\frac{\pi}{\lambda Z} \nabla^{-2} \nabla \cdot \left[\frac{\nabla^{-2} \nabla \Delta I(\mathbf{r})}{I(\mathbf{r}, 0)} \right], \quad (5)$$

where ∇^{-2} is the inverse Laplacian operator that, in the momentum representation, is

$$F[\nabla^{-2}] = \frac{1}{4\pi^2 q^2}. \quad (6)$$

To bypass the alignment steps between the three images needed for a TIE reconstruction, we replaced one of the

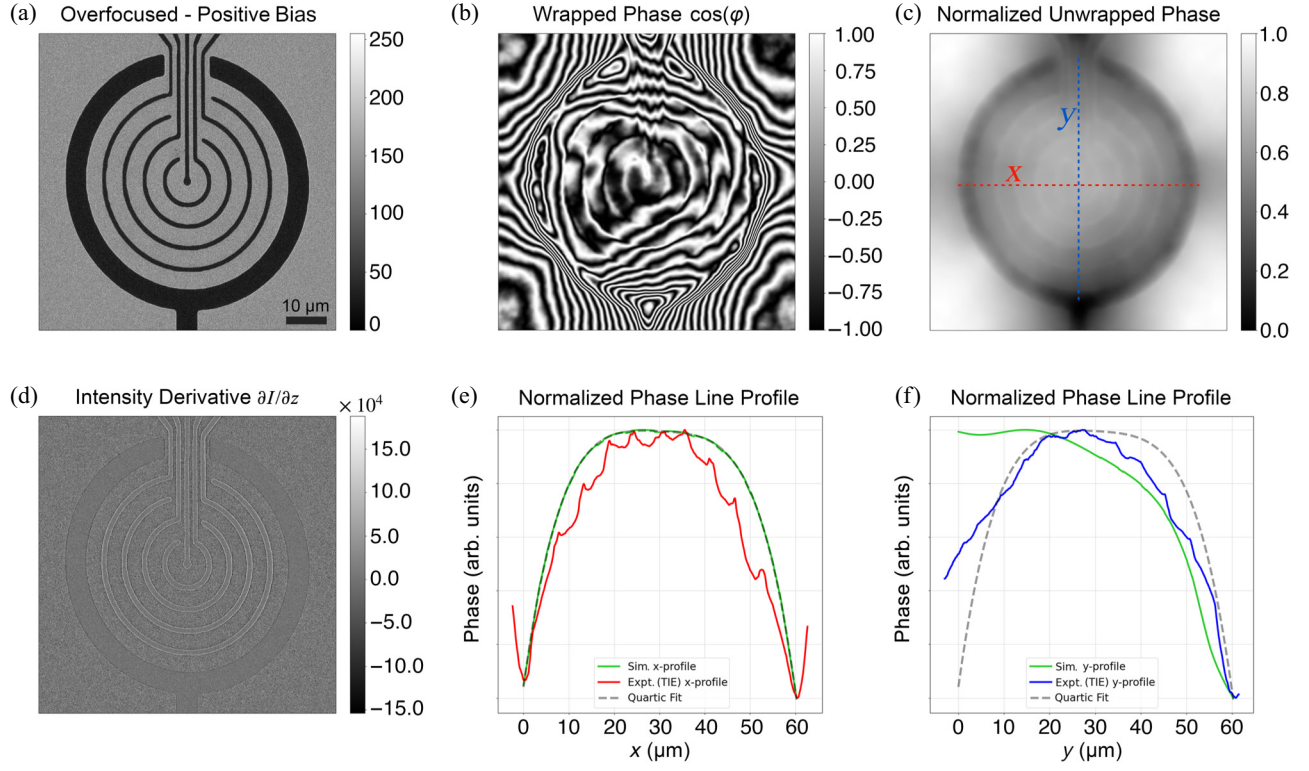


FIG. 6. Transport of intensity equation-based phase reconstruction. (a) Overfocus transmission electron microscope images (400-μm defocus); (b) wrapped phase; (c) normalized unwrapped phase map showing x (red) and y (blue) central axes. (d) Calculated intensity derivative $\partial I/\partial z$. (e) Comparison of x -direction central line profiles for the normalized unwrapped phase (red), simulated values (green), and the target quartic fit (gray). (f) Comparison of y -direction central line profiles for the normalized unwrapped phase (blue), simulated values (green), and the target quartic fit (gray).

two defocused images with the image of the device at the opposite bias. In the small defocus limit, bias and defocus are interchangeable. In fact, inverting the bias turns φ into $-\varphi$, and therefore ψ into ψ^* . As detailed in Ref. [32], the TIE equation derives from the interference between the object wave and its Laplacian (representing Fresnel propagation over small distances),

$$I(\mathbf{r}, Z, +) = \left| \psi(\mathbf{r}) + \frac{i\lambda Z}{4\pi} \nabla^2 \psi(\mathbf{r}) \right|^2, \quad (7)$$

where the $+$ represents positive bias. Switching $+$ to $-$, we have

$$\begin{aligned} I(\mathbf{r}, Z, -) &= \left| \psi^*(\mathbf{r}) + \frac{i\lambda Z}{4\pi} \nabla^2 \psi^*(\mathbf{r}) \right|^2 \\ &= \left| \left(\psi(\mathbf{r}) - \frac{i\lambda Z}{4\pi} \nabla^2 \psi(\mathbf{r}) \right)^* \right|^2 = I(\mathbf{r}, -Z, +). \end{aligned} \quad (8)$$

This approach, which is very beneficial to the implementation of the algorithm and to the data acquisition stage of the experiments, is valid only in the small defocus limit where Fresnel diffraction does not play a role, thereby maintaining the symmetry between over- and underfocus images. This symmetry is lost once the full Fresnel diffraction kicks in.

Accurate TIE reconstruction requires careful image pre-processing to ensure the accuracy of the results. In principle, raw electron microscopy images contain artifacts, such as noise and outlier pixels, which originate from detector imperfections and factors related to the microscope environment [33]. If these defects are not correctly detected and replaced, they can cause artificial intensity variations, which appear as errors in the phase reconstruction process. Alongside pixel-by-pixel evaluation, we must ensure that all images used in reconstruction share the same intensity for the image background.

Another critical step in preparing the images for the TIE reconstruction is ensuring accurate alignment. TIE requires the comparison between three images: the in-focus image and two defocused images taken with opposite electrical bias. When working with large defocus distances, it is common to observe slight misalignments between the

in-focus and defocused images, such as translational shifts, rotations, or changes in scale. They can disrupt the pixel-by-pixel intensity comparison, leading to errors in the final phase reconstruction.

To prepare the three images for the TIE reconstruction, we developed a PYTHON script utilizing common image processing libraries, including scikit-image (skimage) and SciPy. By evaluating the histogram of pixel intensities, all outlier pixels were detected and replaced with the average of their neighboring pixel values (see SI7 in the Supplemental Material [28]). This correction was performed with particular care to ensure that the overall data remained essentially unchanged, as only a tiny fraction of pixels—fewer than 100 out of approximately 16×10^6 in each 4K-resolution image—required modification.

Next, the two defocused images were normalized with respect to the background intensity using the in-focus image as a reference. Finally, all three images were carefully aligned to correct for any misalignment, again using the in-focus image as the alignment reference. Once the images were prepared correctly and aligned, they were ready for phase reconstruction using the TIE method. The PYTHON scripts used for TIE phase reconstruction are available from the authors upon request.

The simulation results were compared with experimental data obtained via phase reconstruction using the TIE. A series of TEM images was acquired at three defocus conditions—focus, overfocus, and underfocus—with defocus values ranging from approximately 400 μm to 5 mm. For each defocus setting, images were recorded under three biasing conditions: unbiased, positively biased, and negatively biased. These image sets were then processed using the TIE reconstruction algorithm implemented in PYTHON to retrieve the phase distribution of the phase plate. The results of this reconstruction are shown in Fig. 6.

Figure 6 presents the results of TIE-based phase reconstruction. Figure 6(a) shows the overfocus TEM image acquired at a defocus value of approximately 400 μm under positive bias. These images were preprocessed to ensure accurate intensity normalization and alignment. Figures 6(b) and 6(c) display the reconstructed phase maps, presented in both wrapped (cosine of the phase) and unwrapped forms, respectively. The phase maps are normalized to a 0–100 scale for illustrative purposes. This normalization enables straightforward visual comparison between different biasing conditions and across simulations, although any information about the scaling parameter φ_0 in Eq. (3) is lost. Figure 6(d) shows the calculated intensity derivative along the optical axis $\partial I/\partial z$ as defined in Eq. (4).

Finally, Figs. 6(e) and 6(f) show experimental and simulated radial line profiles extracted from the unwrapped phase map along the central x and y axes with a quartic fit overlaid for comparison. Generally, the experimental results follow the expected beam shaping behavior,

although two discrepancies can be noted. First, the experimental profile is less smooth than the simulated one, especially along the x axis. This is attributed to the physical presence of the electrodes, which results in narrow peaks most likely caused by absorption and scattering of the electron beam. Second, the experimental data show much better behavior along the y axis, compared to the simulations, suggesting that the contacts play a less important role than the simulations predicted.

IV. CONCLUSIONS

We designed and fabricated an adaptive thin-film electrostatic phase plate with an active area consisting of gold electrodes supported on a thin SiN_x membrane. This is the first experimental demonstration of an adaptive thin-film electrostatic phase plate for TEM. The fabrication process involved three critical steps, which we optimized to maximize the fabrication throughput. First, we split the silicon etch required for SiN_x membrane opening into two parts: approximately 90% etching before the electrode fabrication and approximately 10% subsequent etching. This was a compromise between maximizing the wafer structural stability and minimizing the etching time after metal deposition. Second, we employed image reversal for electrode patterning to ensure a good lift-off. Finally, we optimized the SiN_x thickness, balancing the requirements for electron beam propagation with stability during fabrication, and we employed a protective resist coating prior to wafer dicing to maximize membrane survival. Our fabrication process resulted in 174 chips per wafer, with a yield of approximately 95% chips with intact active areas after dicing.

To assess the functionality of our phase plates, we first ran simulations to identify the appropriate potentials required to produce a quartic beam phase profile, i.e., where the beam phase is proportional to the fourth power of distance from the center. Simulations showed that the asymmetry of the electrode layout given by the electrical contacts is expected to result in bilateral symmetry of the resulting phase profile, instead of the desired radial symmetry. Experimental Fresnel imaging data using the biased phase plate as a TEM sample was compared with the simulation data from COMSOL with an identical biasing profile. The normalized unwrapped reconstructed phase retrieved experimentally shows comparable behavior to the profile expected from the simulations. Along the central x axis, the reconstructed phase approaches the desired quartic profile. There is an asymmetry along the central y axis, where the electrode contacts introduce a phase shift. This phase shift, however, appears less prominent in the experimental data than in the simulations.

Future work should focus on improving the smoothness and symmetry of the active area phase profile. A smoother phase profile should be achieved by making the electrodes

thinner or narrower. The symmetry of the phase profile may be improved by topology optimization. This would imply training a model to predict which electrode layout would be optimal for spherical aberration correction and using the model to produce new electrode patterns for the active area of the phase plate. When the desired phase profile is experimentally demonstrated, the performance of the phase plate for its intended function, i.e., for spherical aberration correction of the electron beam, should be tested.

This work establishes a robust protocol for fabricating a thin-film adaptive electron phase plate. While alterations to the design are necessary before the device can be employed for its intended purpose of spherical aberration correction, the fabrication process itself can easily accommodate design changes in the electrode pattern. Thus, our results represent a valuable step towards the development of adaptive electron optics. The fabrication process is scalable, allowing for the production of around 165 viable chips on a single 100-mm wafer.

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The authors declare no competing interests.

DATA AVAILABILITY

The data that support the findings of this article are openly available [34], embargo periods may apply.

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