APPLICATION NOTE

LARGE SCALE FRESNEL ZONE PLATES ON SiN-MEMBRANES

by

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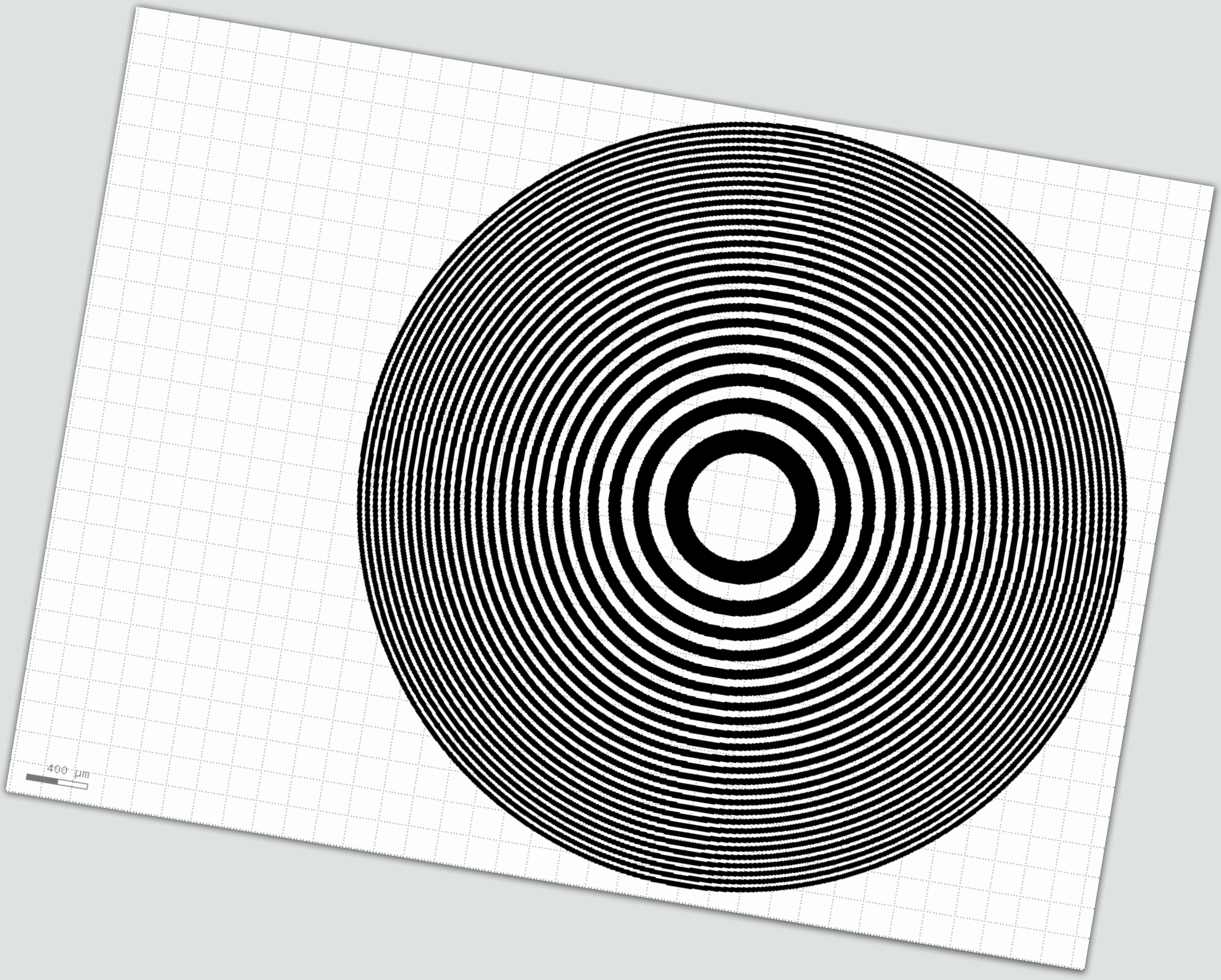

Fresnel Zone plates, using diffraction, are commonly used to focus X-rays. They consist of many concentric zones that get narrower with increasing radii. The ring-shaped zones alternate between two different materials usually with more opaque and more transparent properties, respectively (Fig. 1). Like their refractive cousins, FZPs focus the incident beam into a focal point. The size of the focal point is determined by the smallest zone width on the outside of the zone plate.

For X-ray optic, the opaque zones of the FZP must be made of more absorbing material such as gold or nickel. In order to to keep the general absorption low, a very thin membrane of silicon nitride (typically between 50 nm and 2 µm thick) is used as substrate on which the structures will be built.

Handling and processing membranes can be challenging since they are highly sensitive to mechanical forces. The thin silicon nitride membrane is suspended onto a silicon frame (Fig. 2). This ensemble is henceforth called "membrane chip". In this application note, we present the process established by XRnanotech to structure thick photoresist coated on the membrane. In this example a largescale Fresnel-Zone-Plate (FZP) is fabricated within the resist layer by polymerization using direct-laser-writing.

Figure 2: Schematic of a membrane chip. The silicon nitride membrane (green) is suspended and hold by the silicon frame (blue).

MOUNTING

Many tools in a cleanroom use clamps or vacuum chucks to hold standard substrates in place. If the frame of the membrane chip is large enough, clamps can be placed on the frame without damaging the membrane. But vacuum chucks would bend or break the membrane. Thus, a membrane chip using only a large frame is not suitable. We developed a mounting procedure which can be used with various substrate holding technologies clamping or vacuum. In this case, we decided to use a 4-inch wafer to hold the membrane-chip.

Two silicon chips are bonded on a 4-inch silicon wafer (Fig. 3). The membrane chip being suspended between those two chips is fixed to them with a solvent-soluble material.

The suspension allows the membrane to "breath" when exposed to heat e.g. during the soft-bake of the photoresist. It is also important to place the membrane chip close to the wafer center to get a uniform layer with spin-coating.

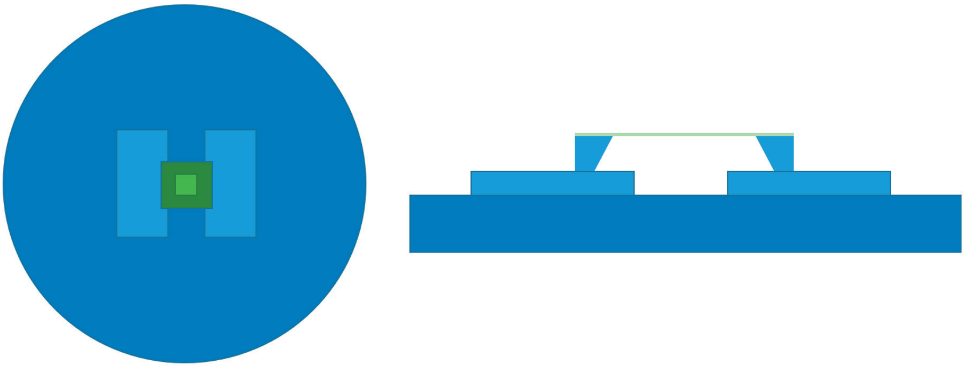

Figure 3: Schematic of the mounting approach: the membrane chip (light blue and green) is suspended between the silicon chips (light blue) that are fixed on the silicon wafer (blue)

Figure 1 on front page: Fresnel Zone Plate design

RESIST COATING

The membrane chip mounted on a 4-inch wafer can be processed with standard tools: the 4-inch wafer chuck of the spin-coater and the hotplate. We also found that using a slow temperature ramp for the soft bake helped to avoid thick edge beads covering a large area and detaching of the resist.

To fabricate the FZP, we used the mr-DWL5 resist, a negative tone photoresist from Microresist Technology GmbH, optimized for 405nm exposure. The spin-coating parameters for the desired resist thickness (11µm) were given by the manufacturer's datasheet. Only the baking step was modified with a longer ramp to reduce the edge bead.

EXPOSURE

The FZP design was created with python and exported in gds format. The smallest feature was 25 µm wide. A direct laser writer, a DWL 66$^+$ by Heidelberg Instruments, was used to expose the photoresist coated membrane. The ensemble (membrane chip on a 4-inch wafer) was held with vacuum onto the stage of the direct laser writer.

The DWL 66+ offers two possibilities to keep the laser beam focused during the exposure, a pneumatic autofocus system that uses a flow of compressed air and an optical autofocus using a red laser. The latter was used because the flow of air of the pneumatic autofocus would have bent the membrane. Once the write head was focused, we moved to one of the corners of the membrane using the linear stage and the camera image. Since there is a significant difference in material thickness between the membrane (maximum 2 µm) and its frame (hundreds of micrometers), the suspended area of the membrane is easily identifiable in the camera image as it will appear much darker than the frame. Using the corners as reference, the design could be exposed perfectly in the center of the suspended membrane.

The exposure being made with a focused laser beam, it also brings heat to the photoresist that it exposes. If the laser power is too high, the heat can induce stress into the membrane, and it could potentially break. This rarely happened but, when high doses are required, we suggest using a multi-pass exposure to reduce the focused laser energy at a given time. This can easily be done by increasing the exposure count parameter of the direct writer while reducing the laser energy used for exposure.

DEVELOPMENT

A post exposure bake is necessary to cross-link the mr-DWL resist. For this baking step, the temperature suggested by the resist supplier can be used, a slower ramping temperature step is not necessary. Afterwards, the sample must rest in order to rehydrate. Then, the membrane chip can be unmounted from the wafer because it will not be exposed to high temperature and air pressure variations anymore.

The development of the sample can be performed like any other sample: placing it into a developer bath and gently agitating it. It is recommended to use a holder, for example a basket with holes, to protect the membrane.

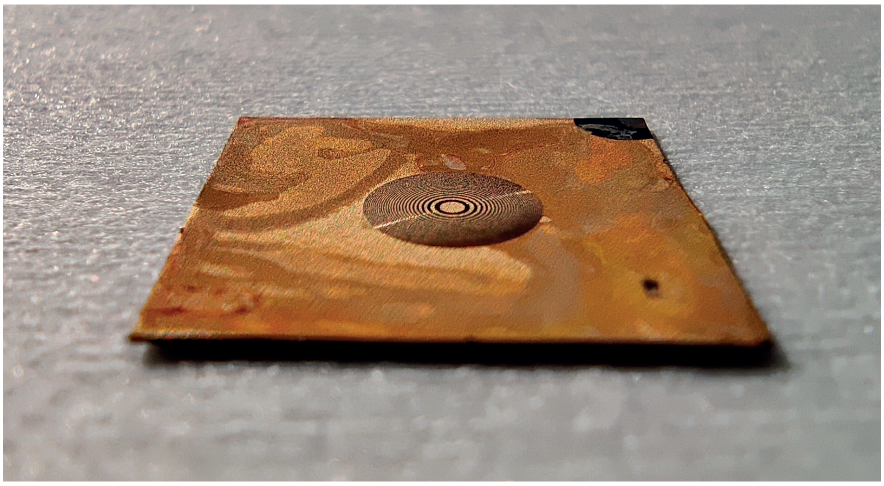

Figure 4: Membrane chip with gold plated Fresnel-Zone-Plate. The dark circles are the photoresist, which was exposed using the WMI of the DWL 66$^+$

CONTRAST MATERIALS

In many cases a larger contrast between the absorbing and transmitting rings will lead to a better Fresnel Zone Plate efficiency. For some applications, the contrast between air and photoresist can be sufficient, but in our case, we increased the contrast by replacing the air with gold, the resist becoming the transmitting material (Fig. 4). A 10 µm high gold layer was grown on the sample to fill the non-exposed rings. The gold having a much higher absorption than the photoresist. In case of low x-ray energies, it would be necessary to remove the photoresist to increase the transmissivity further.

RESULTS

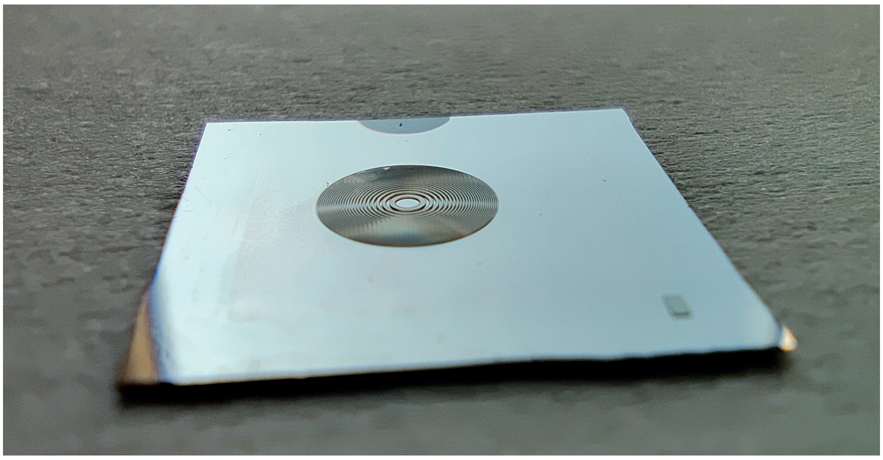

Figure 5: Membrane chip with plating base and laser written FZP on top (made out of photoresist). The resist height is approx. 30 µm and was fabricated as a prototype for taller zones.

Examples of Fresnel Zone Plates fabricated with this methodology can be found in the following publications:

Full-field x-ray fluorescence imaging using a Fresnel zone plate coded aperture: https://doi.org/10.1364/OPTICA.477809

Colour images from the shadow of a sample: https://www.uni-goettingen.de/en/3240.html?id=6961

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