

Investigation of Metal-Assisted Chemical Etching for Fabrication of Silicon-Based X-Ray Zone Plates

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X-ray zone plates are diffractive focusing optics widely used in x-ray microscopes. The zone plate pattern is a circular grating structure with decreasing line widths towards its outer part. The width of the outermost zone defines the resolution of the zone plate, whereas the thickness defines the focusing efficiency. Fabrication of high-resolution and high-efficiency zone plates are challenging due to the limitations in achievable aspect ratios. Metal-assisted chemical etching (MACE) is a wet-chemical method used for micro- and nano-patterning of silicon (Si) and fabrication of high aspect ratio structures [1]. The MACE reaction is catalyzed by a noble-metal, such as gold (Au), which is transferred into the Si through a local electrochemical process. The Au pattern design will determine the morphology of the processed Si and the MACE reaction conditions the quality of the transferred pattern into the Si. MACE has become an alternative to dry etching methods for fabrication of Si-based hard x-ray zone plates [2,3,4]. In the present study, we investigated the impact of etching solution composition, temperature and reaction time in MACE for fabrication of high-quality Si-based zone plate structures [5].

For fabrication of zone plates, p-type Si (100) wafers (1-5 Ωcm) were coated with a resist double-layer of 70 nm poly(methyl methacrylate) (PMMA) and 40 nm ZEP7000. A zone plate design with a diameter of 60 μm , a line-to-space ratio of 1:1 and zone widths ranging from 1 μm to 100 nm was patterned using electron beam lithography. Further, the exposed PMMA and ZEP7000 resist layers were developed in isopropanol/methyl isobutyl ketone and hexylacetate, respectively. Thin layers of 1.5 nm titanium and 25 nm Au were evaporated and the resist layers lifted-off resulting in only the Au zone plate pattern. After lift-off, the sample surface was cleaned using oxygen/argon plasma as preparative step for MACE processing of zone plates. The MACE experiments were performed in a bath of etching solution with varying etching solution composition and temperature (Table 1). The etching time was varied for investigation of achievable etch depths.

The quality of the MACE processed zone plates is determined by many experimental parameters such as catalyst design, reaction kinetics, etching time and Si type, to name a few [1]. We found that the Au pattern defining the zone plate design has a big influence on the etching direction. Addition of interconnects in the zone plate design between the Au rings resulted in vertical and homogenous etching for every investigated MACE condition (Figure 1a). Thus, no electron hole balancing structures were necessary to obtain vertical etching as has been proposed before [2,3]. Increasing the oxidant concentration (hydrogen peroxide, H_2O_2) from 0.17 to 1.4 M, while keeping the etchant concentration (hydrofluoric acid, HF) constant, resulted in deeper etching with smooth and stable zone walls up to a H_2O_2 concentration of 0.68 M (Figure 1b). The MACE reaction was found to be imbalanced at the highest investigated H_2O_2 concentration resulting in rough zone walls. Similarly, processing temperatures above room temperature resulted in rough zone walls. Processing temperature lower than room temperature resulted in slower etching and no effect on the zone plate quality was observed. MACE time series was performed between 4 and 32 min using a H_2O_2 concentration of 0.68 M and room temperature processing.

A constant etching rate of $0.7 \mu\text{m}/\text{min}$ was found for all investigated processing time points. The exact thickness of the zone plate can thus be tuned by the MACE processing time.

Table 1. Investigated MACE process parameters for zone plate fabrication

[HF]:[H ₂ O ₂] (M:M)	Temperature (°C)	Time (min)
4.7: 0.17	25	4
4.7:0.34	25	4
4.7:0.68	10, 25, 40	4, 8, 16, 32
4.7:1.4	25	4

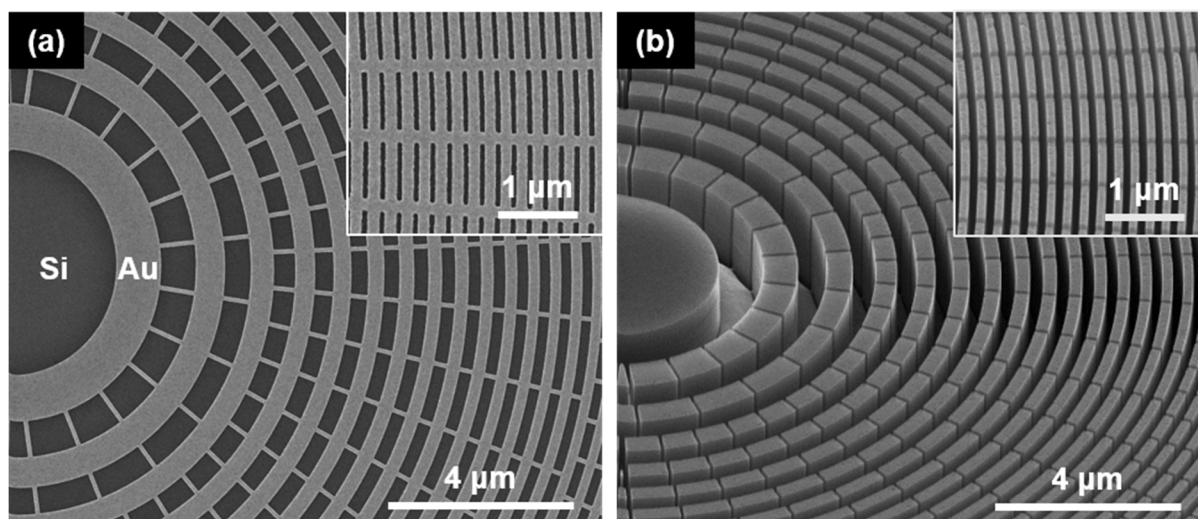


Figure 1. SEM micrographs of (a) Au patterned Si wafer (top view) and (b) MACE processed Si zone plate at [HF]:[H₂O₂]=4.7 M:0.68 M at room temperature for 4 min (52° -tilt view). Insets show the outermost zones.

References

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