EPJ Web of Conferences **287**, 05003 (2023) https://doi.org/10.1051/epjconf/202328705003
EOSAM 2023

Fabrication of nanometre sized gratings via ion irradiation

Johannes Kaufmann[1,*], *Frank* Schrempel[1], and *Uwe* Zeitner[2]

[1]Friedrich-Schiller-University Jena, Institute of Applied Physics, 07745 Jena, Albert-Einstein-Str. 15, Germany
[2]Fraunhofer Institute for Applied Optics and Precision Engineering, 07745 Jena, Albert-Einstein-Str. 7, Germany

Abstract. The damage caused by irradiation of crystalline material with ions results in localized volume changes. Here, swelling is utilized to fabricate nanostructured gratings with heights below 10 nm for extreme ultraviolet radiation. Irradiations were performed through a structured layer of photoresist shadowing parts of the sample from a broad ion beam. This enabled much shorter fabrication times than comparable direct write processes with a focussed ion beam. The study presents results from first systematic investigations regarding the fabrication of nanostructured gratings by irradiation of silicon with a broad beam of helium ions with energies of 30 keV. A smaller, scanned beam is used for comparison. Fluence was varied from 0.4 to 7.5×10^{16} $ions/cm^2$. Fabricated structures were measured via atomic force microscopy. This yielded a controllable method to fabricate shallow gratings with heights in the range of 0 to 10 nm.

1 Introduction

The precise fabrication of nanostructured gratings with a vertical dimension below 10 nm is of great relevance for applications in the EUV and X-Ray range. This can be achieved with localized swelling due to ion irradiation. It is already clear that direct write processes are feasible, but impractical for large areas [1,2]. To maintain applicable processing times, irradiation with a broad beam is necessary. For a broad beam previous experiments utilized stencil masks to selectively shadow parts of the irradiated surface [3]. However, this approach required lateral grating sizes of at least 10 µm and had large transition regions between shadowed and irradiated areas. Some ion target combinations even exhibited self-organisation and did not require any mask [4]. To their detriment, such approaches are limited in their structural freedom. To avoid these issues, we utilized a resist mask to shadow parts of a broad beam of helium ions. For those the amorphization and formation of helium bubbles in silicon is already well known [5]. To expand on the limited range of parameters shown in previous studies we will pay special attention to the swelling created by different fluences of ions, but also look into the effects of flux and energy variation. The application of multiple similar fluences gives insight into the reproducibility and controllability of our method.

2 Experimental procedure

The study uses superpolished Si-(100) wafers provided by SILTRONIC AG as substrate. The mask is a binary lamellar structure with a period of 1 μm and a fill factor of 0.5. Figure 1 shows a scanning electron microscope (SEM) image from a cut through the mask. A layer of aluminium oxide and AZ-1505 photoresist masks part of the sample. The resist was structured in an electron beam lithography process utilizing chromium and reactive ion beam etching. Samples were irradiated with helium ions accelerated to energies between 20 and 55 keV. The fluence was varied from 0.4 to 7.5×10^{16} $ions/cm^2$. To investigate the effects of different irradiation conditions the experiments where performed at two ion beam sources. The source 4GABIS of the Institute of Applied Physics Jena is a specialized broad beam ion source with a target diameter of up to 150 mm. It irradiates the whole sample with a flux of 2×10^{14} $ions/cm^2/s$ [6]. The more conventional ion source ROMEO of the Institute of Solid State Physics Jena scans a beam with a mm-diameter across the sample. This results in an average flux of 3×10^{12} $ions/cm^2/s$. The samples were clamped to the target without further thermal contacting. The variation of ion energies was conducted at ROMEO.

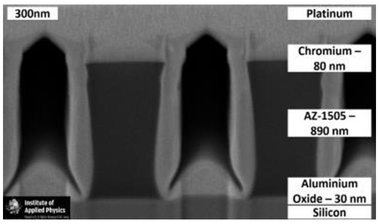

Fig. 1. SEM image of a cut through the utilized mask. The platinum on top is a residual of the FIB-cutting process.

3 Results and discussion

The representative structure shape depicted in fig. 2a shows that the swelling results in a sine like height profile. This is to be expected due to a mixture of strain relaxation and plastic flow of the material, and the distribution of

---

[*] Corresponding author: johannes.michael.kaufmann@uni-jena.de

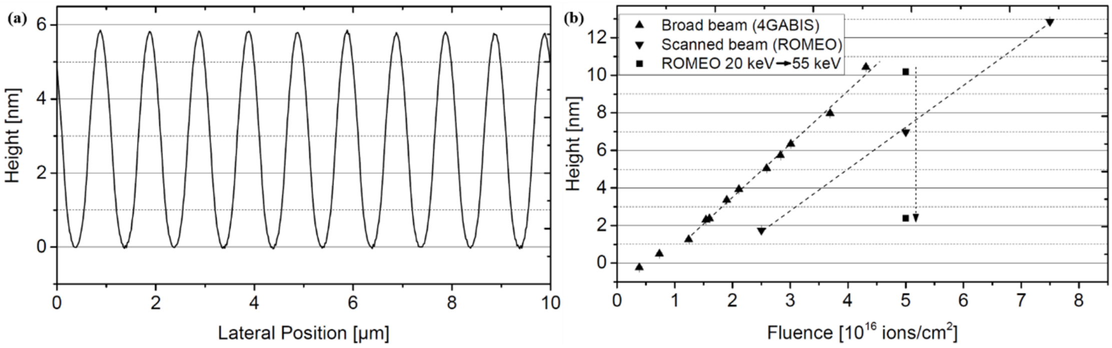

Fig. 2 (a) Example of the profile shape as measured by AFM. **(b)** Height in dependence of fluence for the helium ion irradiations performed at 4GABIS and ROMEO. All data marked with a triangle was measured on samples irradiated with $30\ keV$ helium ions. The dashed lines are guides to the eye. The dotted arrow indicates the change for an increase of ion energy from 20 to $55\ keV$.

helium ions inside the silicon [2]. The swelling caused by ion irradiation is uniform across neighbouring trenches in the mask. All heights are measured as the difference between high and low points of the observed profiles.

The change of measured structure height with fluence of helium ions shown in Fig. 2b indicates a close to linear relation over large parts of the investigated range for both the irradiations with sources 4GABIS and ROMEO, although the experiments at the latter resulted in a lower height for the same fluence. Heights ranging from below 0 to 12 nm were observed. Multiple experiments with similar fluence conducted at 4GABIS yielded results closely following the same relation. This indicates a great reproducibility of the observations made here.

The observed swelling results from a mixture of sputtering, point defect, and bubble formation during the irradiation, the latter having the strongest impact here [1,5,7]. With increasing fluence a larger amount of helium proportionally increases the volume of the bubbles and resulting swelling. Volume changes related to point defects are deemed minor in comparison to the ones caused by the bubbles. This also explains the observation of a negative height step into irradiated areas for the lowest fluence applied here. In this case, the sputtering at the surface outweighs the volume changes introduced by point defects, whereas the fluence is still below the threshold for a formation of bubbles.

The difference between the results from the two ion sources is presumed to be related to the higher irradiation temperature at 4GABIS, which originates from the higher flux. A higher temperature increases the ability of silicon interstitials to migrate to the surface, where they are observed as a height step [8]. Adapting the provided equations, the maximum of swelling for a given fluence would be expected for an irradiation temperature of ~700 °C. This is above the 500 °C measured as equilibrium irradiation temperature for the irradiations conducted at 4GABIS. Due to the lower ion flux, the irradiation temperature at ROMEO will be also smaller, which can explain the lower measured height.

That the increase in ion energy lead to a lower height can have at least two origins: (a) at higher energies larger lateral spread of helium ions leads to more swelling in the regions shadowed from direct impacts. This would only decrease the measured height, while the absolute swelling of irradiated areas would remain unaffected. (b) The formation of helium bubbles in deeper regions of the lattice could affect the migration of silicon interstitials and the relaxation of lattice strain, shifting away from the surface and into the bulk. This carries a reduction in the absolute swelling at the surface of irradiated areas. To further investigate this, additional measurements with e.g. transmission electron microscopy are necessary.

4 Conclusion

The investigations of silicon irradiated by helium have yielded a quick, controllable and reproducible method to fabricate shallow nanostructured gratings required in EUV and X-Ray optics. It was shown that next to the fluence, irradiation temperature and energy are also parameters greatly impacting the structure height. Further research will look into possibilities to affect the structure shape via irradiation conditions and post-processing.

The authors thank Patrick Hoffmann from the Institute of Solid State Physics of the FSU Jena for the operation of ROMEO.

References

1. X. Huang, Y. Xie, M. Balooch, S. Lubner, P. Hosemann, J. Appl. Phys. **132**, 025106 (2022)
2. G. García, M. Martin, M. D. Ynsa, V. Torres-Costa, M. L. Crespillo, M. Tardío, J. Olivares, F. Bosia, O. Peña-Rodríguez, J. Nicolas, M. Tallarida, Eur. Phys. J. Plus **137**, 1157 (2022)
3. Y. Zhou, S. Li, Y. W. Q. Huang, W. Zhang, Y. Yao, J. Hao, Y. Sun, M. Tang, B. Li , Y. Zhang, J. Hu, L. Yan, Carbon **148**, 387-393 (2019)
4. J. Li, G. Yang, R. M. Bradley, Y. Liu, F. Frost, Y. Hong, Nanotechnology **32** 385301 (2021)
5. K. E. Haynes, *Defect Evolution During Elevated-Temperature Helium Ion Implantation into Silicon*, University of Florida (2019)
6. D. Tang, S. Pu, Q. Huang, H. Tong, X. Cui, P. K. Chu, Nucl Instrum Methods Phys Res B NUCL INSTRUM METH B **257**, 801-804 (2007)
7. L. Pelaz, L. A. Marqués, J. Barbolla, J. Appl. Phys. **96,** 5947-5976 (2004)
8. S. Tamulevičius I. Požėla, M. Andrulevičius, Mat. Sci. and Eng. **B40**, 141- I46 (1996)