Test procedure for locally assessing potential risks of sputter damage due to thruster plumes

IEPC-2015-156／ISTS-2015-b-156

*Presented at Joint Conference of 30th International Symposium on Space Technology and Science,
34th International Electric Propulsion Conference and 6th Nano-satellite Symposium
Hyogo-Kobe, Japan
July 4–10, 2015*

Markus Piechotka, Kristof Holste, Torsten Henning,* Klaus Hannemann, and Peter J. Klar
Institute of Experimental Physics I, Justus Liebig University, DE-35392 Giessen, Germany

Vladislav V. Nigmatzyanov and Andreas Neumann
German Aerospace Center (DLR), DE-37073 Göttingen, Germany

An important issue in the use of thrusters on a satellite is the need to minimize the interaction of the plume with components of the satellite such as solar panels, antennas etc. Part of the danger in case of ion thrusters is the bombardment of the components with atomic particles of the neutralized beam which may, as a primary effect, cause material erosion by physical sputtering and, as a secondary effect, redeposition of the sputtered material on the components. Both may lead to malfunctions such as short-circuits or opaque contamination of solar panels etc. We have designed microstructured test chips for probing the effects of erosion in the plume of an ion thruster which may be used as add-ons in terrestrial lifetime testing in large vacuum chambers at distances comparable with the dimensions of satellites. The test chips are 5 mm×5 mm in size on a silicon substrate. A regular pattern consisting out of titanium microstructures, which serve as etch masking during ion bombardment, i.e. protect the underlying silicon in the etching process, is defined on the surface of the test chip by lithographic methods. Removing the remainder of the titanium after the test chip has been exposed to the ion beam and removed from the vacuum chamber allows one to study the height differences between silicon areas which were protected by titanium in the etching process and those directly exposed to the ion bombardment. Such depth profiles are easily obtained by AFM or stylus profilometer measurements and allow one to determine the etch depth into the silicon with great accuracy and with a high lateral spatial resolution and thus to assess the damage caused by the thruster plume. This test chip design is very versatile and can be easily extended to erosion studies of other materials used for components of satellites.

Nomenclature

Symbols

I	initial beam current at extraction orifice
r	distance from the extraction orifice along the beam direction
$j(r)$	average beam current density at duistance r from the extraction orifice
Δt	duration of exposure in the thruster plume
Δh	etch depth caused by ion impact
α	divergence angle of the thruster beam
R_0	initial beam radius defined by the extraction orifice
$R_1^{0.95}(r)$	beam radius at distance r from the extraction orifice

*corresponding author, Torsten.Henning@physik.uni-giessen.de

Abbreviations and acronyms

AFM	atomic force microscopy
EP	electric propulsion
SC	spacecraft

I. Introduction

It is an objective of electric propulsion engineering to minimize the interaction of the thruster plume with the spacecraft, since the particles in the plume, whether still charged or already neutralized, will have a detrimental effect on the spacecraft's surfaces. Primarily, they will cause sputtering of the surfaces, resulting in material erosion. Additionally, redeposition of sputtered material may lead to undesirable coating of parts of the spacecraft. Most at risk are windows (not only for the visible wavelength range), antennas, and solar arrays. To avoid such detrimental EP/SC interaction, the thruster beam has to have a low divergence angle, with a sharp drop of the particle density beyond a maximum opening angle, and any sensitive elements of the spacecraft will have to be placed as far away from the plume as possible. However, due to collisions with the ion optical system or due to interactions in the beam, a certain fraction of the ejected particles will always be on a collision course with spacecraft surfaces. The assessment of EP/SC interaction is largely carried out by simulation calculations. Such simulation models,[1–6] however, will have to be verified against measurements of the actual erosion of surfaces in ion beams with known beam parameters. We propose to place standardized erosion test chips in and around the beam/plume when carrying out beam diagnostics measurements in the usual large vacuum tank facilities to gather information about the sputter erosion and, if applicable, material redeposition that can then be correlated with the characteristics of the beam/plume and the position of the test chip with respect to the thruster. Such chips would have to be small in size not to disturb the diagnostics measurements simple in their use, yet provide enough versatility to explore a number of different surfaces that are of real importance to spacecraft engineering.

One approach would be to coat the chips with a thin film of known thickness and measure the remaining film thickness after the chip has been exposed to the beam for a certain time. This would, however, have to rely on very precise measurements of the film thickness, and the film thickness would have to be tailored to the expected erosion depth before the vacuum chamber is closed for the test run. Instead, we propose to measure the step height profile resulting from the erosion when the surface of interest has partially been covered by another material, and this material has been removed. Prerequisites for this technique are, first, that the masking material is not completely removed during the exposure to the beam, and that, second, the masking material can be removed selectively from the material of interest, that is without removing the material of interest.

There are several combinations of masking material and material whose erosion is to be measured that very well fulfill these requirements. One example, that we shall concentrate on in the following, is silicon as the material of interest and titanium as the masking material. Silicon, in the form of a single crystalline wafer, is a very well defined material. Once the native oxide has been removed, silicon wafers with the same crystal orientation will produce identical results independent of any issues with vendor, production method or production batch. Titanium has one of the lowest sputter erosion rates of all materials, and it can be removed from the silicon surface with very high selectivity by wet chemical etching with hydrofluoric acid ("HF dip").

The step heights after reasonable exposure times can be expected to lie in the micrometer or sub-micrometer range. Such step height can be measured by stylus profilometers or by scanning probe microscopes, namely by atomic force microscopes (AFM). AFM will not only deliver the step height, but additonally yield information about the surface morphology, e. g. the roughness, after the erosion process. AFM instruments have a limited field of view, typically less than 100 micrometers squared. In order to have more than one data point per measurement, a number of steps should fit on the 100 micrometer square. Therefore, the masking material should be patterned by microfabrication techniques.

II. Sample preparation

Here the additive processing in the microstructuring process was performed using titanium as masking material and 2-inch silicon wafer as probe material. The corresponding process flow is depicted in Fig. 1.

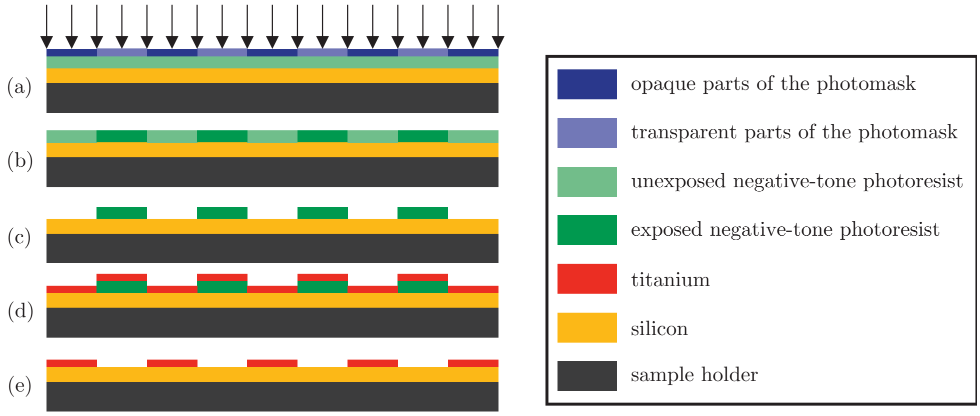


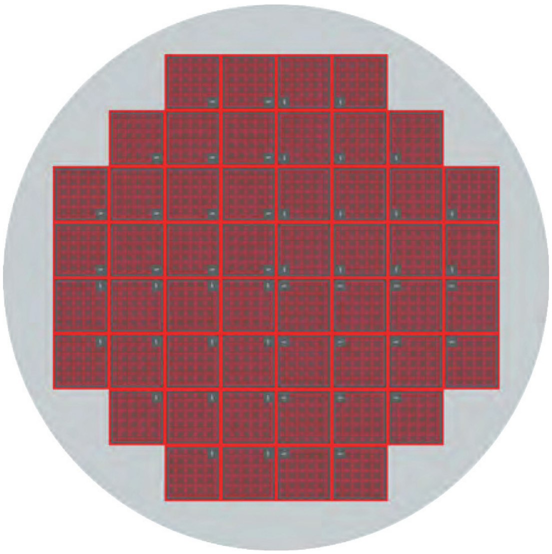
Figure 1: Process flow of the sample preparation.

The silicon wafer coated with negative-tone photoresist is exposed partially to UV light with the help of a photomask (Fig. 1a). The resist in the areas exposed to UV light is photo-chemically modified, i.e. the pattern is transfered into the photoresist (Fig. 1b). In the proceeding development step, the unexposed parts of the resist are dissolved in the developer, leaving areas of exposed resist with an undercut profile on the substrate (Fig. 1c). Titanium is deposited by thermal evaporation onto the sample, covering the surfaces of the resist and of the silicon areas not covered by (Fig. 1d). In the evaporation process, the edges of the resist are not coated due to their undercut profile. In the final liftoff process step the samples are rinsed in a solvent which dissolves the resist and removes it together with the titanium layer on top of it whereas it does not affect the titanium which is directly deposited on silicon. This results in a defined titanium pattern on the silicon surface (Fig. 1d).

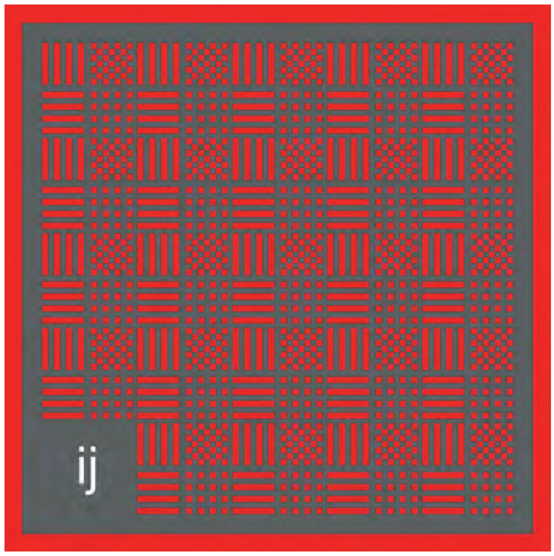
The mask pattern which was transferred onto the 2-inch silicon wafers is shown in Fig. 2a. It consists of a repetition of a well defined substructure defining the actual test chips. The 52 identical substructures are arranged in quadrants on the wafer. Each substructure has a lateral dimension of $5 \times 5\,\mathrm{mm}^2$ and is labeled. The labelling denotes its position on the wafer (Fig. 2b), if the wafer is used as a whole, or serves as an identifier of the test chip, if various test chips are positioned at different locations within a vaccuum chamber during a test of a thruster. A single substructure consists of 24 identical fields ($1 \times 1\,\mathrm{mm}^2$), each comprising four test arrays. The first test array consists of an arrangement of vertical lines, the second of an arrangement of horizontal bars, the third of a chess-board like arrangement of squares, and the fourth of an arrangement of isolated squares. The smallest structures on the test arrays have lateral dimensions of 15 µm or a period of 30 µm and are schematically shown in Fig. 2c. Images taken by scanning electron microscopy with the sample tilted by 40° show details of these arrangements. They demonstrate a good lateral definition and the defined height of titanium structures (Fig. 2d).

III. Experimental setup

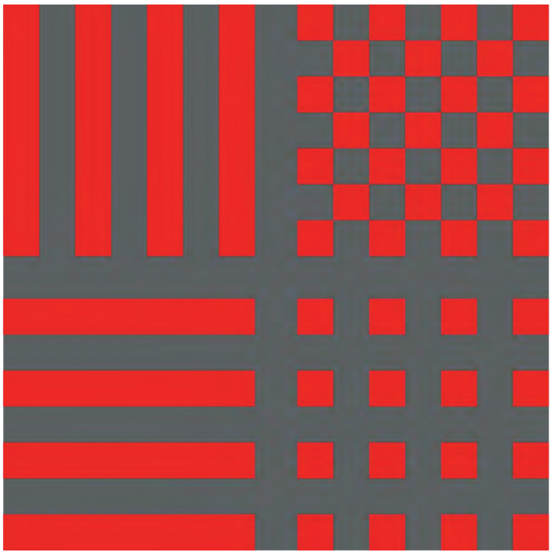
In order to verify the performance of the standardized test chips, down-scaled experiments were carried out using a small vacuum chamber and a RIM-4 thruster with a single extraction orifice. The thruster is based on the radio-frequency ion-thruster (RIT) concept. Figure 3 schematically depicts the experimental setup with the ion thruster and the sample holder in a vacuum chamber. The idea of the experiment was to place the entire wafer with its arrangement of test chips into the ion beam extracted from the thruster. This allows us to detect spatially resolved across the wafer the erosion caused by the ion beam and to correlate these results with the beam profile determined by emittance measurements at the location of the wafer. Within the chamber the RIM-4 emits Xe^+-ions via the single extraction orifice and a two-grid-system onto a sample holder placed at 40 mm (in the experiments shown here) or 115 mm away from the exraction orifice. At these distances the wafer with its test chips basically samples the entire beam cross section. The ions hit the wafer locally with ion densities corresponding to the beam profile of the thruster and sputter particles from the surfaces of both the exposed silicon and the titanium masking. The titanium mask should be thick



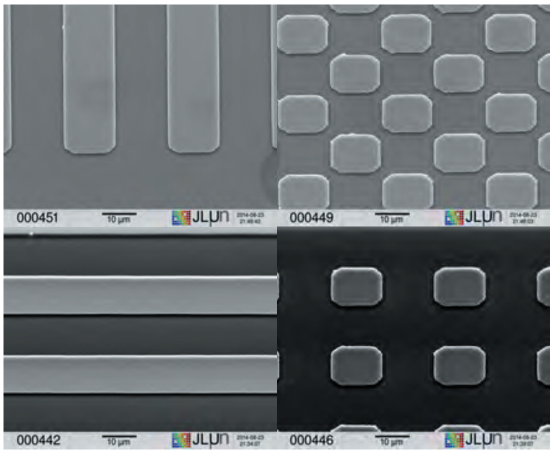
(a) Schematic top view of a structured 2-inch silicon wafer.



(b) Zoom-in of a substructure (test chip) of Fig. 2a with a lateral dimension of 5 millimeters squared (schematic).



(c) Zoom-in of a test chip of Fig. 2b with a lateral dimension of 1 millimeters squared, schematically showing the four kinds of test arrays.



(d) SEM images of the titanium structures shown schematically in Fig. 2c, the SEM images correspond to the four test arrays shown in Fig. 2c.

Figure 2: Mask pattern for the standardized etching procedure.

enough not to be entirely eroded during the measurement time and thus to sufficiently protect the underlying silicon surface areas, which will act as reference levels in the depth profiling. The adequate thickness, of course, depends on several parameters such as the beam parameters (extraction voltage, ion mass, ion charge, ion flux density), the sputter time and the strength of the interaction between impinging ions and masking material (Xe^{+}-Ti-interaction in this case). If the titanium mask is not entirely eroded, the remainder may be removed by selective wet-chemical etching exposing the original silicon surface again. Thus, the depth into the probe material with repect to the original surface may be determined locally and correlated with the beam parameters. The titanium on silicon may be selectively stripped off by using hydrofluoric acid (HF) which etches titanium very fast and does not affect the silicon surface. The depth profiles may be obtained

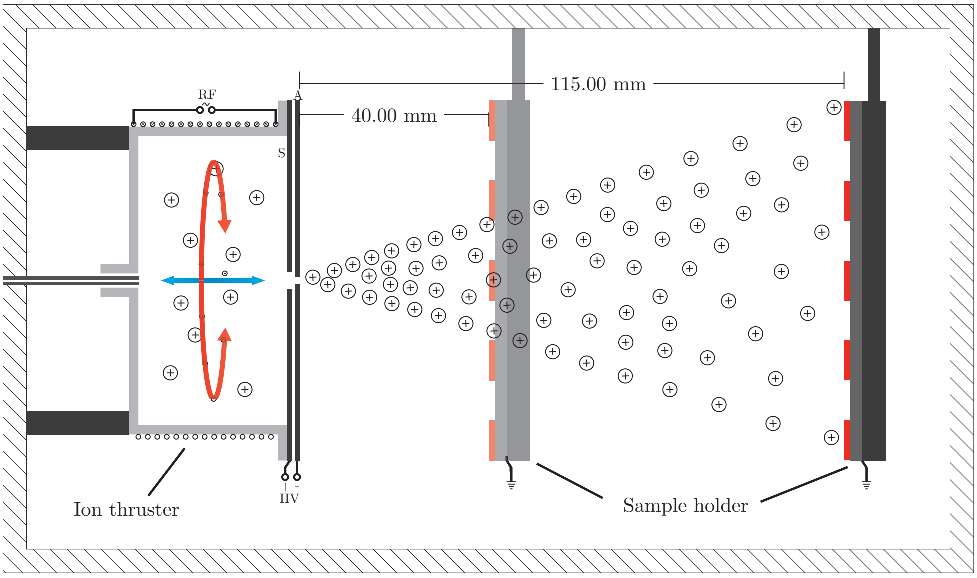
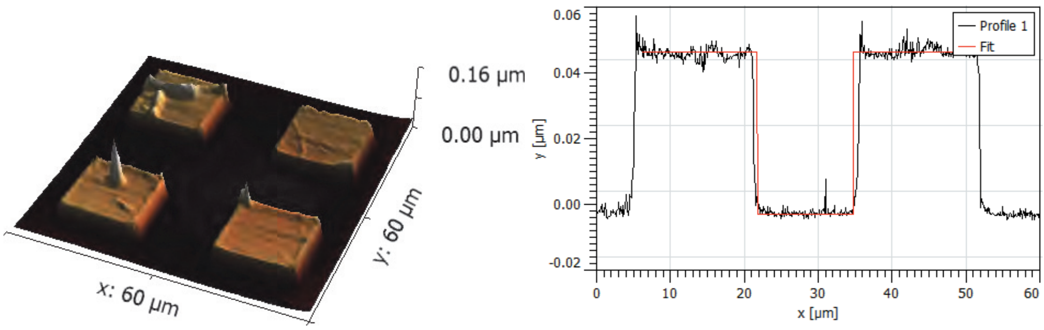


Figure 3: Experimental setup in a vacuum chamber comprising the RIM-4 Xe^{+}-ion source and a sample holder at either 40 mm or 115 mm distance from the accelerator grid opening.

by using an AFM or a stylus profilometer.

IV. Results and discussion



(a) (b)

Figure 4: (a) 2D AFM profile scan of parts of an arrangement of individual squares after exposure to ions of the RIM-4 Xe^{+}-ion source. The titanium masking layer was removed by HF-etching prior to the AFM measurements. (b) Depth profile extracted from the 2D AFM profile depicted in (a).

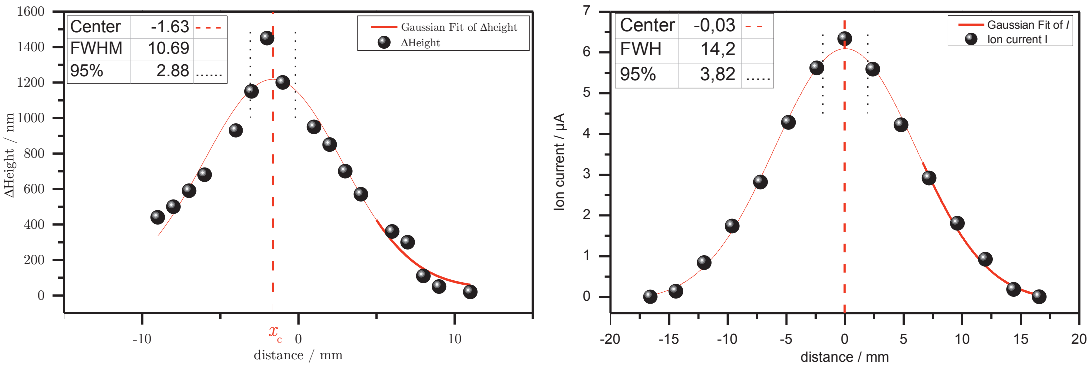
The erosion experiments were performed with the patterned 2-inch silicon wafer positioned in front of the extraction orifice at a distance of 40 mm and centered with respect to the thruster axis. The thruster used Xe as propellant and emitted ions with an energy of 1500 eV, at an average current density of 61 $\mu A/mm^2$ at the extraction orifice. The patterned wafer was exposed to the ion emission for 3 hours. Afterwards it was removed from the vaccuum chamber and the remainder of the titanium was stripped off the silicon prior to determining of the depth profile across the wafer. Local information can be obtained by an AFM analysis by two-dimensional AFM profile scans in areas of up to 100 µm × 100 µm. An example of such a two-dimensional AFM profile scan of a test pattern consisting of individual squares is shown in Fig. 4(a). The AFM image reveals that the square pattern as depicted in the lower right image in Fig. 2c is indeed transfered into the silicon wafer. The individual squares are clearly visible. One-dimensional depth profile

Table 1: Erosion test parameters.

distance from grid orifice	40 mm	115 mm	7 m
ion energy	1500 eV	1500 eV	1500 eV
ion current	174 µA	116 µA	1 mA
extraction orifice diameter	1.9 mm	1.9 mm	1.9 mm
sputter time	3 h	3 h	2000 h
maximal erosion depth	1450 nm	70 nm	350 nm

across individual or a few structures of the pattern yield the local etch depth at this position, in this case 49 nm (Fig. 4(b)).

One-dimensional scans using a stylus profilometer are better suited for probing a wider scan range. The measured depth values extracted from the profilometer scans are found to be in good agreement with the values extracted from two-dimensional AFM scans. The profilometer scans are performed in constant force mode. We performed profilometric measurements at positions distributed along a straight line passing through the center of the silicon wafer (which coincided with the center of the ion beam in the erosion experiment described above). The extracted depth profile as a function of position on the wafer is depicted in Fig. 5a.



(a) (b)

Figure 5: (a) Erosion depth profile in silicon after the exposure to Xe^+ ions from a RIM-4 on a standardized silicon/titanium test chip placed 40 mm away from the ion source. The beam center is 1.6 mm off the centerline. (b) Corresponding beam profile of the RIM-4 recorded 40 mm away from the ion source. The beam center could be assigned to be on the centerline. The beam width (95 % of beam intensity) is 3.8 mm. The extraction parameters are given in the text.

The erosion profiles were compared with the beam profile. A beam profiles can be obtained by performing an emittance measurements in a plane perpendicular to the ion beam axis.[7] Figure 5b shows such a beam profile obtained at a distance of 40 mm from the extraction orifice, where the silicon wafer was located during the erosion experiment. The erosion depth profile shown in Fig. 5a and the corresponding beam profile exhibit both an almost Gaussian shape, with full-width-at-half-maximum of 10.7 mm and 14.2 mm, respectively. The positions of the profile centers also almost match and are only 1.6 mm apart. Thus, a strong correlation between the two profiles is established.

Based on the results obtained in our small vacuum chambers with the test chip close to the ion source and the crude approximation given below, we suggest that the proposed titanium/silicon test chip design is suitable for studying erosion effects in the outer regions of a plume of an ion thruster, i.e. at distances several meters away from the thruster. One possible application would be the use of the test chips as an add-on in a lifetime test of thrusters in larger vacuum chambers such as JUMBO in Giessen or STG-ET in Gttingen which much better correspond to the situation occurring on a satellite in space in terms of distances between thruster and solar panels etc.

Assuming a beam divergence α and an initial beam current I at the accelerator grid with an initial beam radius R_0, the average beam current density at a distance r from the accelerator grid will be $j(r)$:

$$j(r) = \frac{I}{\pi \left[\tan(\alpha)\, r + R_0\right]^2}. \tag{1}$$

In a very simple approach, the average etch rate $\frac{\mathrm{d}h(r)}{\mathrm{d}t}$ is proportional to the average ion density $j(r)$, i.e. the etch depth $\Delta h(r) \propto j(r) \cdot \Delta t$ where Δt is the exposure time. Thus, for constant α and R_0 and constant beam parameters, but the average current I itself, the ratio of erosion depths $\Delta h_i = \Delta h(r_i)$ at different distances r_i is given by

$$\frac{\Delta h_2}{\Delta h_1} = \frac{I_2 \Delta t_2}{I_1 \Delta t_1} \left[\frac{\tan(\alpha)\, r_1 + R_0}{\tan(\alpha)\, r_2 + R_0}\right]^2, \tag{2}$$

where I_i and Δt_i denote the initial beam current and the duration of exposure, respectively, in the situations compared. In our experiment, the beam divergence may be estimated from the beam width (95 % of the beam intensity) $2R_1^{0.95}$ of the experiments performed at a distance of 40 mm:

$$\tan(\alpha) = \frac{R_1^{0.95} - R_0}{r_1}, \quad r_1 = 40\,\mathrm{mm}, \tag{3}$$

yielding $\alpha = 3.4°$ where the mean value of both the beam widths from the erosion depth profile and the beam profile was used for $R_1^{0.95}$, i.e. 2.9 cm and 3.8 cm, respectively, cf. Fig. 5. Based on this value and the experimental parameters of the erosion test performed at a distance of 40 mm (cf. Tab. 1), the erosion depth of a structure in an experiment with a different exposure time, initial beam current and sample distance can be extrapolated using Eq. (2). For example, an erosion depth of $\Delta h_2 = 350\,\mathrm{nm}$ in silicon is estimated for a sample exposed for $\Delta t_2 = 2000\,\mathrm{h}$ at a distance of $r_2 = 7\,\mathrm{m}$ from a thruster emitting $I_2 = 1\,\mathrm{mA}$ Xe^{+}-ions with 1500 eV. Such an etch depth lies well in a suitable range for the probes used for determining the depth, i.e. stylus proliometer measurements or AFM measurements. Considering that the etch rate of the titanium masking layer is lower than that of silicon, a thickness of the titanium layer of a few hundred nm is sufficient to ensure that the titanium is not entirely removed in the erosion process. As the erosion process is rather a physical, but a chemical process, it is a reasonable assumption that the erosion effects due to neutralized beams like in a real thruster yield similar results in terms of etching rates than our preliminary tests.

V. Conclusion

We have designed microstructured test chips 5 mm×5 mm in size on a silicon substrate for probing the effects of erosion in the plume of an ion thruster. The silicon test chips are covered with regular patterns consisting out of titanium microstructures which serve as etch masking during ion bombardment, i.e. protect the underlying silicon in the etching process. Removing the remainder of the titanium after the test chip has been exposed to the ion beam and removed from the vacuum chamber allows one to study the height differences between silicon areas which were protected by titanium in the etching process and those directly exposed to the ion bombardment. The depth profiles obtained by AFM or stylus profilometer measurements allow one to determine the etch depth into the silicon with great accuracy and with a high lateral spatial resolution. Estimates show that these test chips are suitable add-ons in lifetime tests of ion thrusters. Furthermore, this test chip approach can be easily extended to erosion studies of other materials used for components of satellites in the vicinity of ion thrusters. Therefore, such studies will yield valuable information in the context of EP/SC interaction.

Acknowledgments

This work was supported in the framework of LOEWE-Schwerpunkt RITSAT. Samples were made at the Micro- and Nanofabrication Laboratory at Justus Liebig University Giessen.

References

[1] Yan, L., Wang, P., Ou, Y. H., and Kang, X. L., "Numerical Study of Hall Thruster Plume and Sputtering Erosion," *Journal of Applied Mathematics*, 2012, pp. 327021.

[2]Passaro, A., Vicini, A., Nania, F., and Biagioni, L., "Numerical Rebuilding of SMART-1 Hall Effect Thruster Plasma Plume," *Journal of Propulsion and Power*, Vol. 26, No. 1, 2010, pp. 149 – 158.

[3]Tajmar, M., Sedmik, R., and Scharlemann, C., "Numerical Simulation of SMART-1 Hall-Thruster Plasma Interactions," *Journal of Propulsion and Power*, Vol. 25, No. 6, 2009, pp. 1178 – 1188.

[4]Nadiradze, A. B., Obukhov, V. A., and Popov, G., "Numerical Rebuilding of SMART-1 Hall Effect Thruster Plasma Plume," *Acta Astronautica*, Vol. 64, No. 9 - 10, 2009, pp. 979 – 987.

[5]Tajmar, M., Gonzalez, J., and Hilgers, A., "Modeling of spacecraft-environment interactions on SMART-1," *Journal of Spacecraft and Rockets*, Vol. 39, 2001, pp. 393–399.

[6]Likar, J. J., Bogorad, A. L., Malko, T. R., Goodzeit, N., Galofaro, J. T., and Mandell, M. J., "Interaction of charged spacecraft with electric propulsion plume: On orbit data and ground test results," .

[7]Holste, K., Schippers, S., Müller, A., Spädtke, P., Smirnova, M., and Feili, D., "Emittance measurement setup facilitating plasma diagnostic of ion thrusters," *The 33rd International Electric Propulsion Conference. The George Washington University, USA. October 6–10, 2013.*, 2013, IEPC-2013-191).