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Variations of milling parameters and their effect on the volumetric sputtering yield of Silicon with the use of a plasma focused ion beam

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ABSTRACT

Volumetric sputtering is a technique used to polish the surface of materials at the atomic level used in the production of semi-conductors and nanotechnology. This study examined the effects of varying milling parameters, specifically voltage and current, on the volumetric sputtering yield of silicon using a plasma focused ion beam with argon gas. The experimental process used a Helios 5 Hydra DualBeam Scanning Electron Microscope Plasma Focused Ion Beam (SEM PFIB) to mill silicon surfaces with various voltage-current combinations. Milled depths were analysed in ImageJ, and volumetric sputtering yields were further calculated. The results demonstrate a statistically significant difference between the effects of voltage and current on the volumetric sputtering yield (ANOVA $p = 0.02$). Current was identified as a significant factor ($p = 0.008$), while voltage alone did not show statistical significance ($p = 0.395$). Accelerating voltages of 12 kV and 30 kV demonstrated the most stable and efficient sputtering processes. The results concluded that the inverse proportionality, $Y_s = \frac{V_{\text{removed}}}{q}$, was present between volumetric sputtering yield and charge, through volume removed, with a significant correlation between their natural logarithms ($p = 0.003$).

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YASJ Editors' Note: Reviewers commended the author's creative approach to measurement and communication. They asked to highlight that in longer term studies it would be traditional to include sequential measurement and the determination of uncertainty. However, this novel approach represents a thorough starting point for further studies.

INTRODUCTION

Plasma Focused Ion Beam (PFIB) milling is used in a range of industries that require high precision levels such as semiconductor production and nanofabrication (Höflich et al., 2023) (Bassim et al., 2014). PFIB milling is used to produce nanometer thin sections of biological specimens for in depth structural and compositional analysis in dental research (House et al., 2022). The rise of advanced technologies, such as artificial intelligence and quantum computing, has further expanded the applications of PFIB milling. The development of next generation silicon-based microchips and nanodevices is reliant on PFIB milling for the precise etching and patterning of optoelectronic nanoscale structures. Furthermore, recent advancements in gas source ion beams (GSIBs), such as He+ PFIBs, offer the potential to replace Ga+ PFIBs due to their ability to achieve nano-resolution imaging with lower implantation damage and pollution (Li et al., 2021).

Volumetric sputtering refers to the amount of surface material removed per unit of electric charge. It involves the ejection of atoms or ions from a

material's surface, typically a metal, caused by the bombardment of energetic ions. Sputtering occurs when these incoming ions transfer energy to the atoms in the solid, leading to the ejection of atoms from the surface. The volumetric sputtering yield can be mathematically expressed as the following :

$$Y_s = \frac{V_{\text{removed}}}{q} \tag{1}$$

Where Y_s is the volumetric sputtering yield, V_{removed} is the volume of the material removed, and q is the charge delivered, expressed in units of $\mu\text{m}^3/\text{nC}$ (Kim et al., 2012).

Milling is a technique defined as the process of removal of material from the surface of a substrate, in this case, with the use of a focused ion beam. *Milling parameters* refers to the variables that govern and affect the outcome of how much material is removed from the substrate. Voltage and current are the two parameters investigated in this study.

The effect of high voltage on the volumetric sputtering yield

High voltages increase the energy of the bombarding ion and are responsible for strong electric fields, consequently increasing the ion acceleration; leading to efficient sputtering and a high sputter rate.

The ion has sufficient momentum to cause a continuous cascade of momentum transfer in the target material (Hashmi, 2016). Therefore, a number of surface atoms within the target will be disturbed and accelerated out of the material. These ejected atoms will travel in a straight line until they deposit on a nearby surface in an inelastic collision. As high voltages increase the ion energy, the collision rate increases, increasing the sputter rate as a result. Refer to Figure 1.

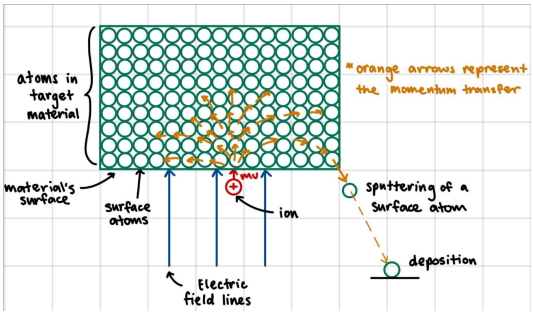


Figure 1. Ion bombardment of a target material, showing sputtering and deposition. The diagram is not to scale.

The effect of beam current on the volumetric sputtering yield

High currents result in a greater flow of charged ions impacting the target material within a given time period. As the current increases, the number of ions striking the target material per second also increases, leading to a high collision rate that results in the ejection of surface atoms from the target material. Consequently, the volumetric sputtering yield increases with beam current, which can be mathematically described as:

$$I = \frac{q}{t} \tag{2}$$

where I is current, q is charge, and t is time.

Despite the extensive interest in gallium ion milling in literature (Kim et al., 2012) (Pastewka et al., 2009), other ion species, such as argon, appear to have been comparatively understudied. While singly-charged gallium ions (Ga+) ions have been the focus of numerous investigations due to their effectiveness in various milling applications, the exploration of alternative ions remains limited.

Silicon was chosen as the material of study due to its importance in semiconductor technology and its extensively reported response to ion beam processing, making it an ideal material for evaluating the effectiveness of different ion species and milling parameters, further providing methods to optimise PFIB processes to establish the scope of upcoming microelectronics (Siew et al., 2021).

This study addresses the understudied areas within the field by using argon as the ion species in a plasma focused ion beam system to examine how varying milling parameters, specifically voltage and current, affect the volumetric sputtering yield of silicon. Understanding the effects of these parameters will contribute to the development of more effective and versatile ion beam tools, leading to advancements in semiconductor manufacturing and other high precision applications.

It is hypothesised that the volumetric sputtering yield of silicon in argon PFIB milling is more strongly influenced by beam current than the accelerating voltage, given the direct dependence of material removal on ion flux and accumulated charge. To examine this relationship, the effects of systematically varying beam current and accelerating voltage on volumetric sputtering yield are investigated under controlled milling conditions.

METHODOLOGY

Preparation of the silicon sample

The silicon sample was prepared through a cleaning process, beginning with 2 minute sonication in acetone to remove contaminants. This was followed by a second sonication in isopropyl alcohol (IPA) to ensure thorough cleansing. Finally, the sample was dried using pressurised nitrogen gas, leaving it free of any remaining residues. This process was completed to reduce the likelihood of any unwanted particles, such as dust or oils, on the silicon surface, which may interfere with the experimental procedure and affect the accuracy of the results.

Preparation of the scanning electron microscope plasma focused ion beam

Calibration of the SEM PFIB (Helios 5 Hydra DualBeam) takes place overnight at the end of each week. This is completed so angle measurements are accurate and to reduce magnification errors (Ghosh, 1975).

Process of imaging with the scanning electron microscope

The Helios SEM was operated through the use of an algorithm, written in Python and automated to mill each area of the silicon sample with specific voltage - current conditions (Table 1). The full code is provided in Supplementary Code S1. The collection of one set of sputtering yield data took place overnight (4 - 8 hour period).

Process of analysis using ImageJ

Each image was uploaded into the free image-analysis software ImageJ. The software was calibrated and used to find the depth of the image by counting each selected pixel. After the depth was found, mathematical calculations were carried out to determine the correct milled depth. The full ImageJ analysis procedure and an example calculation of the corrected milled depth are provided in the supplementary material.

Table 1. Milling depths for silicon using argon ions (n=1). An en dash indicates that a value was unavailable.

Voltage (kV)	Current (nA)	Milled depth (µm)	Volumetric sputter yield (µm^3 nC^{-1})
5	0.2	–	–
5	3.1	0.4962	0.5335
5	4.8	0.7348	0.5103
5	9.3	0.7868	0.2820
5	20.0	0.7690	0.1282
8	0.2	–	–
8	3.9	0.4442	0.3796
8	6.1	0.5812	0.3176
8	9.5	0.4784	0.1679
8	20.0	0.8211	0.1368
10	0.21	–	–
10	1.4	0.0850	0.2024
10	5.9	0.4442	0.2509
10	12.0	0.8211	0.2281
10	35.0	1.1459	0.1091
12	0.21	–	0.0000
12	1.2	0.1371	0.3807
12	4.8	0.5304	0.3684
12	14.0	1.1193	0.2665
12	50.0	2.2576	0.1505
30	0.2	–	–
30	0.7	–	–
30	2.0	0.1536	0.2559
30	7.6	0.4619	0.2026
30	28.0	2.0012	0.2382

RESULTS

Figure 2(a) shows that as charge increases, volumetric sputtering yield decreases for each voltage. This is consistent with the inverse relationship suggested by Equation (1), where $Y_s \propto \frac{1}{q}$. Although the volume removed is not constant, the overall hyperbolic trend remains evident. Localised deviations from this trend are observed at specific data points, including (840, 0.2382) at 30 kV and (177, 0.2509) and (360, 0.2281) at 10 kV, where the volumetric sputtering yield increases rather than decreases.

Figure 2(b) shows that milled depth increased with charge for all voltages. Higher voltages resulted in greater milled depth for equivalent charge, as indicated by the sharp increase in milled depth at 12 kV and 30 kV.

Figure 3 shows the silicon sample after milling under the five accelerating voltages and associated beam-current conditions listed in Table 1.

Statistical analysis

The hypothesis can be restated as null and alternative hypotheses:

H_0: There is no significant difference between the

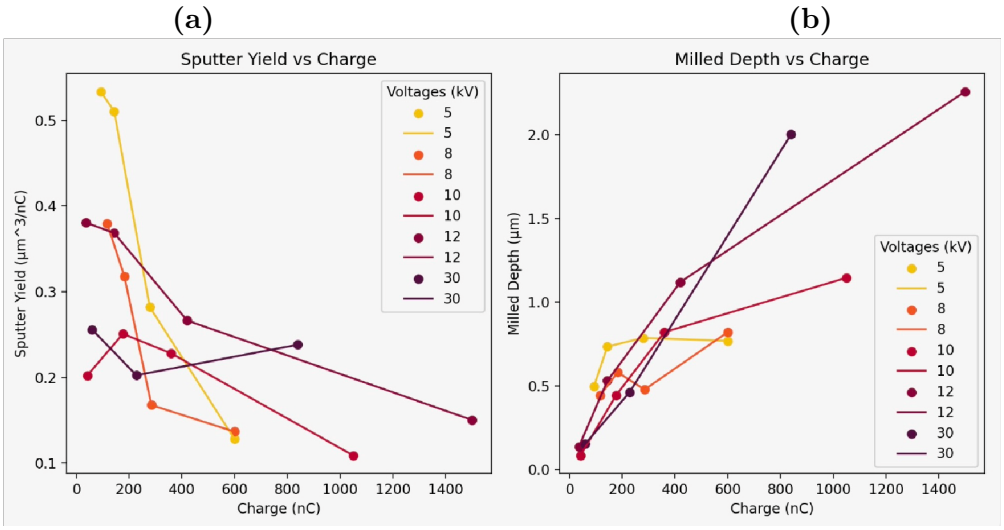


Figure 2. (a) Volumetric sputtering yield of silicon as a function of delivered charge. (b) Milled depth of silicon as a function of delivered charge.

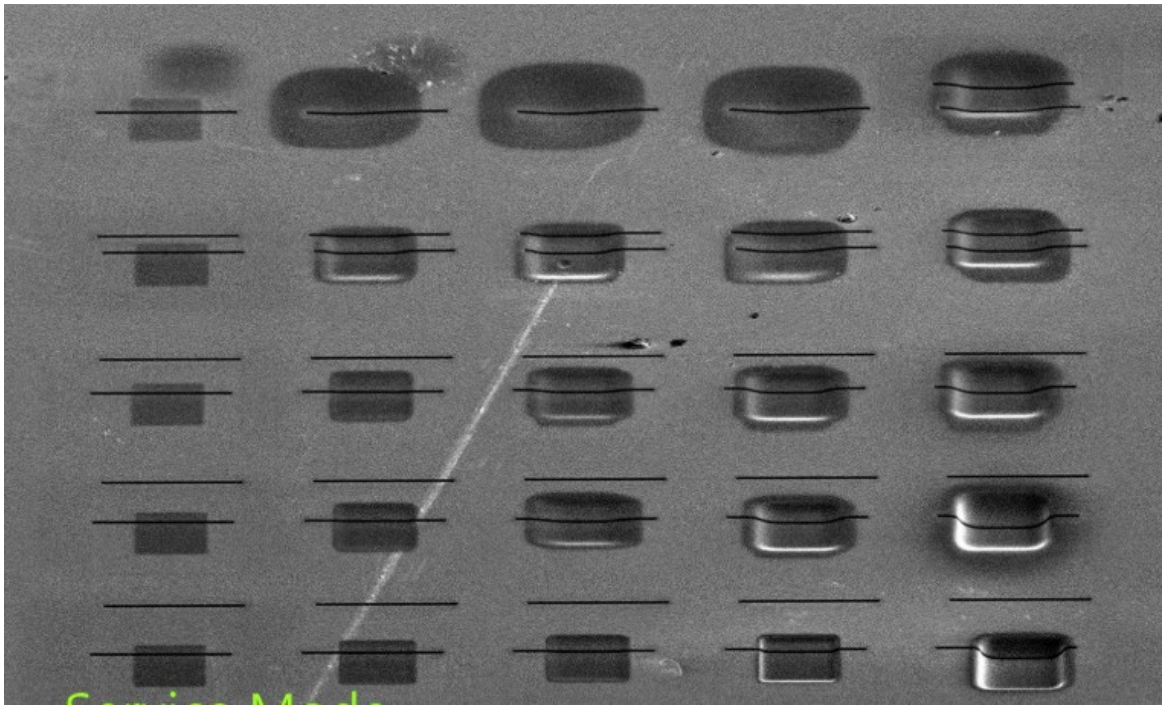


Figure 3. Silicon milled using argon ions. Rows show accelerating voltages of 5, 8, 10, 12 and 30 kV from top to bottom; beam current varies between milled areas (see Table 1).

effect of voltage and current on the volumetric sputtering yield of Silicon.

H_1: There is a significant difference between the effect of voltage and current on the volumetric sputtering yield of Silicon.

The ANOVA significance value was calculated using the multiple linear regression model:

$$Y = \hat{\beta}_0 + \hat{\beta}_1 X_1 + \hat{\beta}_2 X_2 + \epsilon$$

Using the reported units, the fitted multiple linear regression model is:

$$Y_s = 0.36627 - 0.00536I - 0.00247V \pm 0.04697 \quad (3)$$

where Y_s is measured in $\mu m^3/nC$, I is beam current in nA, and V is accelerating voltage in kV.

The null hypothesis was rejected, as the ANOVA returned a significance value of 0.02 ($\alpha = 0.05$) (Table 2), indicating a significant difference between the effect of voltage and current on the volumetric sputtering yield of silicon. Current was a significant factor affecting the volumetric sputtering yield (p = 0.008), while voltage alone

did not show statistical significance (p = 0.395) (Table 2).

Table 2. p-values for variables associated with volumetric sputtering yield and the ANOVA significance for voltage and current.

Y-variable	X-variable	p	ANOVA
Volumetric sputtering yield	Voltage	0.395	0.02 for voltage and current
	Current	0.008	
	Milled depth	0.183	
	Charge	0.008	–
Natural log of volumetric sputtering yield	Natural log of charge	0.003	–

Using the fitted linear relationship from the log-log plot demonstrated in Figure 4,

$$\ln(Y_s) = -0.2778 \ln(q) + 0.1044$$

the volumetric sputtering yield can be expressed as

$$Y_s = 1.1100\, q^{-0.2778}.$$

This relationship suggests a power-law dependence of volumetric sputtering yield on charge, which may be approximated as

$$Y_s \propto q^{-1/3} \quad \text{or} \quad Y_s \propto q^{-1/4}$$

corresponding to Equations (4) and (5), respectively.

$$Y_s \approx \frac{a}{q^{1/3}} \tag{4}$$

$$Y_s \approx \frac{a}{q^{1/4}} \tag{5}$$

Figure 4 demonstrates a linear relationship between the natural logarithm of volumetric sputtering yield and natural logarithm of charge, indicating that the data follows a power-law model. Additional sampling and analysis are required to determine whether Equation (4) or Equation (5) provides the more accurate description.

The R^2 value of Figure 4 is higher than the R^2 value of Figure 5 (42% compared to 34%), suggesting that the log-log representation provides a better fit to the data than the linear scale. The remaining unexplained variance indicates that additional parameters not investigated in this study also have an influence on the volumetric sputtering yield.

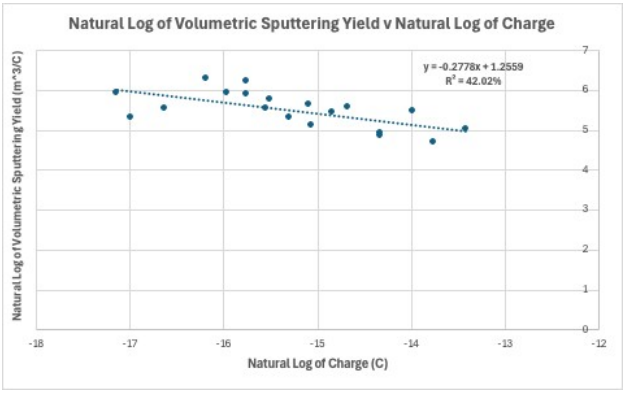


Figure 4. Natural logarithm of volumetric sputtering yield as a function of the natural logarithm of delivered charge.

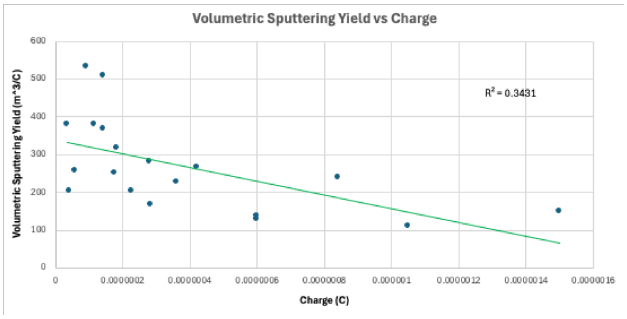


Figure 5. Volumetric sputtering yield as a function of delivered charge, with a fitted trendline.

DISCUSSION

Volumetric sputtering yield and milled depth relationship

Figure 2 shows that the higher the voltage, the more stable and efficient the sputtering and milling processes are, as shown by the measured volumetric sputtering yield and the greater milled depth at high voltages (12 kV and 30 kV).

Inconsistencies in sputtering are shown by the sharp decrease in the volumetric sputter yield value regarding a voltage of 5 kV. This inconsistency can be attributed to redeposition of the sputtered material, surface damage induced by the ion beam, and surface roughening. These factors heavily reduce the efficiency of the sputtering process as milling progresses, leading to low volumetric sputtering yield. The same phenomena have been observed in similar ion beam studies regarding milling of silicon. Prolonged milling introduces complexities and inconsistencies which ultimately decrease the volumetric sputtering yield (Jamaludin et al., 2013).

Further graphical analysis

The volumetric sputter yield generally decreased with increasing charge across all voltages (Figure 2(a)). The rapid drop at lower voltages (5 kV and 8 kV) suggests that the energy provided by the ion beam was insufficient in order to maintain efficient sputtering as charge accumulates. In contrast, the decrease in the volumetric sputter yield was more stable at high voltages as this provided more kinetic energy because the ions carry greater momentum. This allowed them to sustain material removal even as the charge increased, leading to a more stable volumetric sputter yield.

Figure 2(b) shows that high voltages and high charge values ed in a greater amount of material removal, suggesting that they provided enough energy to maintain a high milling rate as charge increased. This may be due to more energetic ions penetrating deeper into the target material, resulting in a more effective milling process.

The SEM PFIB is the industry standard for high precision milling and analysis, ensuring precise and relevant measurements (Kim et al., 2018) (Liu et al., 2020). To minimise potential sources of measurement error, the used SEM was routinely calibrated.

This experiment successfully isolated the effect of the independent parameters, voltage and current, on volumetric sputtering yield by employing argon as the ion species across all milling conditions. The silicon target material and image analysis methodology were kept consistent throughout the study, minimising variability.

The data collected closely aligned with theoretical expectations, with higher accelerating voltages producing more stable volumetric sputtering yields. As shown in Figure 2, the observed trends are consistent with the relationships described by Equation (1), confirming an inverse proportionality between volumetric sputtering yield and delivered charge. Rearranging Equation (1) allows the dependence between milled depth and charge to be expressed as Equation (4), indicating a direct proportionality between milled depth and charge. This proportionality is reflected in Figure 2(b), where the milled depth increased with increasing charge delivered by the ion beam.

$$Y_s = \frac{V_r}{q}$$

where the volume removed is given by $V_r = L \times w \times d_m$.

$$d_m = \frac{Y_s q}{Lw} \tag{6}$$

where d_m is the corrected milled depth, Y_s is the volumetric sputtering yield, q is the delivered charge, and L and w are the length and width of the milled area, respectively.

$$\therefore d_m \propto q$$

Both graphs align with their respective mathematical models, providing initial validation of the observed relationships between charge, volumetric sputtering yield, and milled depth. However, further validation would require a larger sample size in order to reduce statistical uncertainty and improve the statistical confidence of the results.

Variations in the volumetric sputtering yield, particularly at lower voltages, suggest potential sources of uncertainty. These variations may arise from surface roughening, redeposition of sputtered material, and ImageJ-based depth measurement uncertainty. Measurement uncertainty was reduced by using high precision instruments.

The experimental process lacked repetition due to time and resource constraints. The nature of this study required meticulous calibration and execution, as the experimental workflow involved multiple stages, including sample preparation, milling, and imaging. Each session required approximately 4-8 hours for data collection, including overnight processes for imaging and depth measurement with the focused ion beam. The availability of the Helios 5 Hydra DualBeam SEM PFIB was limited, as the instrument was in high demand. Further work would benefit from a large sample size and increased repetition. Despite these limitations, the image analysis methodology remained consistent across all measurements, and the observed trends in each graph aligned with their expected mathematical models.

Future work

Future work should include testing with more ion species, like investigating xenon, nitrogen and carbon, and having larger sample sizes incorporating a broader range of voltage and current values and repeated trials for each ion species. Further analysis of the mathematical models presented in Figures 4 and 5 would also help determine whether Equation (4) or Equation (5) more accurately describes the observed behaviour.

CONCLUSION

This study investigated the effects of varying milling parameters on the volumetric sputtering yield of silicon using a plasma-focused ion beam using an argon ion species. The results demonstrated a significant difference between the effects of voltage and current on the volumetric sputtering yield, with an ANOVA significance value of 0.02 ($p < 0.05$). Higher voltages (12 kV and 30 kV) resulted in more consistent sputtering behaviour, as evidenced by greater milled depths and consistent volumetric sputtering yields. The volumetric sputtering yield showed an inverse relationship with charge, decreasing as charge increases across all voltages. Current showed to be a significant factor, affecting the volumetric sputtering yield ($p = 0.008$), while voltage alone did not show statistical significance ($p = 0.395$). The natural log of charge also demonstrated a significant relationship with the natural log of volumetric sputtering yield ($p = 0.003$), although more work is needed to confirm this relationship. These findings contribute to the understanding of ion beam milling processes and have implications for optimising parameters in semiconductor manufacturing and other high-precision applications which include semiconductor manufacturing, quantum computing, and nanodevice fabrication.

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SUPPLEMENTARY MATERIAL

The supplementary material accompanying this article contains the chemical risk assessment, detailed ImageJ analysis procedure, worked calculations for corrected milled depth and volumetric sputtering yield, Python code used to automate the PFIB milling process, and the full experimental dataset.

The supplementary PDF is available with the online article at https://doi.org/10.67642/g1fw2r66.

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