



**HAL**  
open science

# Elucidating the effects of gas flow rate on an SF<sub>6</sub> inductively coupled plasma and on the silicon etch rate, by a combined experimental and theoretical investigation

Stefan Tinck, Thomas Tillocher, Remi Dussart, Erik Neyts, Annemie Bogaerts

## ► To cite this version:

Stefan Tinck, Thomas Tillocher, Remi Dussart, Erik Neyts, Annemie Bogaerts. Elucidating the effects of gas flow rate on an SF<sub>6</sub> inductively coupled plasma and on the silicon etch rate, by a combined experimental and theoretical investigation. *Journal of Physics D: Applied Physics*, 2016, 49 (38), pp.385201. <10.1088/0022-3727/49/38/385201>. <hal-01374746>

**HAL Id: hal-01374746**

**<https://hal.science/hal-01374746v1>**

Submitted on 3 Jul 2025

**HAL** is a multi-disciplinary open access archive for the deposit and dissemination of scientific research documents, whether they are published or not. The documents may come from teaching and research institutions in France or abroad, or from public or private research centers.

L'archive ouverte pluridisciplinaire **HAL**, est destinée au dépôt et à la diffusion de documents scientifiques de niveau recherche, publiés ou non, émanant des établissements d'enseignement et de recherche français ou étrangers, des laboratoires publics ou privés.



HAL Authorization

**This item is the archived peer-reviewed author-version of:**

Elucidating the effects of gas flow rate on an  $SF_6$  inductively coupled plasma and on the silicon etch rate, by a combined experimental and theoretical investigation

**Reference:**

Tinck Stefan, Tillocher Thomas, Dussart Rémi, Neyts Erik, Bogaerts Annemie.- Elucidating the effects of gas flow rate on an  $SF_6$  inductively coupled plasma and on the silicon etch rate, by a combined experimental and theoretical investigation  
Journal of physics: D: applied physics - ISSN 0022-3727 - 49:38(2016), p. 1-5  
Full text (Publishers DOI): <http://dx.doi.org/doi:10.1088/0022-3727/49/38/385201>

# Elucidating the effects of gas flow rate on an SF<sub>6</sub> inductively coupled plasma and on the silicon etch rate, by a combined experimental and theoretical investigation

Stefan Tinck<sup>1</sup>, Thomas Tillocher<sup>2</sup>, Rémi Dussart<sup>2</sup>, Erik C. Neyts<sup>1</sup> and Annemie Bogaerts<sup>1</sup>

<sup>1</sup>Research Group PLASMANT, Department of Chemistry, University of Antwerp, Universiteitsplein 1, B-2610 Antwerp, Belgium

<sup>2</sup>GREMI, CNRS/Université d'Orléans, Rue d'Issoudun 14, BP 6744, F-45067 Orléans, France

**Abstract.** Experiments show that the etch rate of Si with SF<sub>6</sub> inductively coupled plasma (ICP) is significantly influenced by the absolute gas flow rate in the range of 50-600 sccm, with a maximum at around 200 sccm. Therefore, we numerically investigate the effects of gas flow rate on the bulk plasma properties and on the etch rate, to obtain more insight in the underlying reasons of this effect. A hybrid Monte Carlo - fluid model is applied to simulate an SF<sub>6</sub> ICP. It is found that the etch rate is influenced by two simultaneous effects: (i) the residence time of the gas and (ii) the temperature profile of the plasma in the ICP volume, resulting indeed in a maximum etch rate at 200 sccm.

## I. Introduction

It is generally known that the feed gas flow rate can have a large influence on the plasma characteristics and on the effectiveness of some applications, like in propulsion<sup>1-3</sup> and surface treatment<sup>4-6</sup>. This topic is mostly investigated in the field of plasma jets and torches. The total flow rate determines the species density distribution in these types of plasmas, as well as the shape of the plasma, affecting the applications.

For most other types of discharges that are generated and contained in a specific reactor, the gas flow rate is typically tuned for controlling the plasma shape within the reactor volume.<sup>7</sup> These plasmas

(e.g., capacitively (CCP) or inductively (ICP) coupled plasmas) are also most often used for surface treatment.<sup>8</sup> In the field of microelectronics development, and more specifically for plasma etching, the most popular "tuning knobs" are the gas mixing ratio, operating power, reactor pressure and substrate bias voltage. The gas flow rate typically is chosen so that it is high enough to allow a flux of reactive species towards the wafer, but the latter is usually limited by diffusion rather than by the supply of feed gas. The total gas flow rate is therefore most often considered as a less important process parameter and its effects on the plasma and etch process is not well understood. Nevertheless, it has been shown that the gas flow rate can have a significant effect on the efficiency of the etch system.<sup>9-14</sup>

Monkowski and Boyd reported on an approach for improving etch equipment performances through a high accuracy in situ gas flow monitoring system and control scheme.<sup>9</sup> Lee *et al.* experimentally optimized the etch depth uniformity for plasma etching of large area GaAs wafers using control of the overall feed gas flow rate.<sup>10</sup> Ikegawa *et al.* investigated the effects of gas flow structures on radical and etch product density distributions on wafers in magneto-microwave plasma etching reactors.<sup>11</sup> The same group also performed a Monte Carlo analysis of rarefied gas flow structures and ventilation of etching gas in their reactor.<sup>12</sup> Finally, Kwon and co-workers studied the effects of gas flow rate on the etch characteristics of a low-k SiCOH film with an amorphous carbon mask in dual-frequency CF<sub>4</sub>/C<sub>4</sub>F<sub>8</sub>/Ar CCPs, as well as of silicon nitride with an extreme ultra-violet resist pattern in CH<sub>2</sub>F<sub>2</sub>/N<sub>2</sub>/Ar CCPs.<sup>13-14</sup>

From these studies one can conclude that the gas flow rate is most often tuned for controlling the plasma and etch uniformity on the wafer. However, in some situations, the overall etch rate is also significantly influenced by the total feed gas flow rate, even when the flux of reactive etching species is already diffusion limited. Indeed, as will be illustrated below, we found a clear maximum etch rate at 200 sccm gas flow rate in the range of 50-600 sccm, when etching blanket Si wafers with SF<sub>6</sub> ICPs. Halogen-based plasmas are very suitable for silicon etching, since volatile products are created on the surface of the wafer (i.e., chemical etching), which are easily removed by ion bombardment (i.e., sputtering). Fluorine-based gases like SF<sub>6</sub> entail the highest etch rates on Si because F is the most

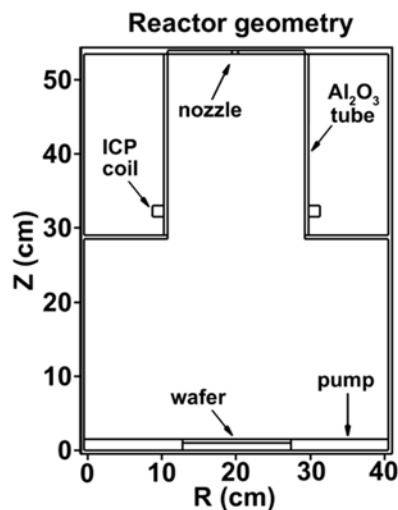
reactive halogen towards silicon, creating volatile  $\text{SiF}_4$ . Indeed,  $\text{SF}_6$ , usually mixed with  $\text{O}_2$ , is a popular gas (mixture) for fast (cryogenic) anisotropic etching of Si.<sup>15-16</sup>

The etching of Si with  $\text{SF}_6$  without  $\text{O}_2$  or any other gas component is relatively simple from a chemical point of view. The  $\text{SF}_6$  gas is fed into the plasma reactor, where it dissociates into reactive products, e.g., F atoms, which entail the high etch rate. It is known that the  $\text{SF}_x$  ( $x = 0-6$ ) species do not contribute significantly to the etching process<sup>17</sup>, so it can be concluded that the chemical etching of Si with F atoms is the only important surface reaction in this system, if no bias would be applied. If a high enough voltage is applied on the substrate electrode, physical sputtering by positive ions also becomes important, next to chemical etching by F atoms.

To understand why in our experiments the etch rate first increases and then decreases as a function of gas flow rate within the range of 50-600 sccm, we have applied a computational model, which can reveal the underlying mechanisms.

## II. Experimental

Si wafers of 15 cm diameter are etched with an  $\text{SF}_6$  ICP in an Alcatel 601E ICP reactor.<sup>18</sup> The geometry of this reactor is illustrated in **figure 1**.



**Figure 1.** Two-dimensional illustration of the ICP reactor used for the experiments.

The SF<sub>6</sub> gas is fed from the top nozzle and the plasma is sustained by the ICP coil surrounding the 20 cm diameter alumina tube powered by an RF power supply at 13.56 MHz. Underneath the source there is a 40 cm diameter diffusion chamber with the wafer at the center bottom. The wafer temperature is kept fixed at 20°C.

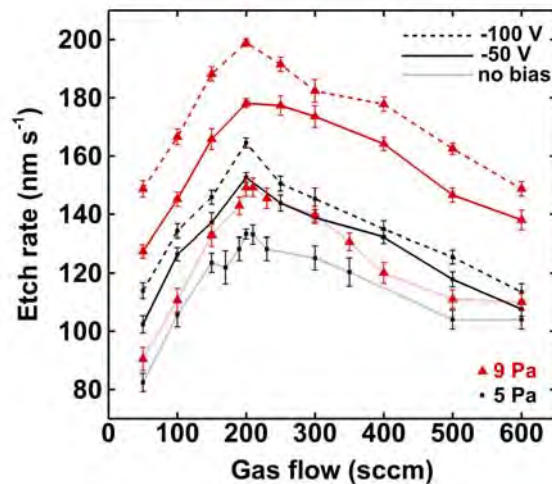
The etch rate is measured in situ with double-point reflectometry, which consists of a 650 nm laser diode from which two separate spots can be positioned at different spots on the wafer: usually a point on the mask and one inside a trench. The interacting phase shift between the two reflected beams is recorded to obtain the etch rate during etching.

### III. Computational details

A two-dimensional hybrid plasma model (called the HPEM), developed by Kushner and coworkers, is applied to predict the plasma characteristics.<sup>19</sup> It combines (i) a module to address the electromagnetic fields by solving Maxwell's equations, (ii) a Monte Carlo module for the electrons and (iii) a fluid module for all other heavy species. With these three modules, the overall SF<sub>6</sub> plasma characteristics can be calculated. A detailed description of the model can be found in this reference.<sup>19</sup> For addressing the SF<sub>6</sub> plasma chemistry, we have adopted the reaction set from Mao *et al.*<sup>20</sup> The model is applied to the reactor geometry illustrated in **figure 1**, at the same conditions as used experimentally.

### IV. Results and Discussion

The Si wafers were etched with pure SF<sub>6</sub> at two different chamber pressures of 5 Pa and 9 Pa for various gas flow rates ranging from 50 sccm to 600 sccm, without bias, and with a bias voltage of -50 V and -100 V. The operating power was fixed for all cases at 1000 W and at 13.56 MHz. The substrate bias voltage was also operated at 13.56 MHz. The measured etch rates obtained for all these conditions are presented in **figure 2**.



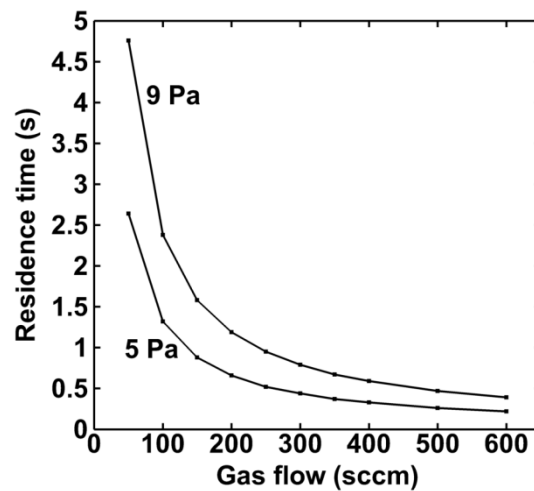
**Figure 2.** Measured etch rates for 5 Pa and 9 Pa, without bias, and with a bias voltage of -50 V and -100 V, as a function of total feed gas flow rate.

The etch rate clearly shows a maximum around 200 sccm for all conditions. The effect is indeed very significant, as the etch rate increases roughly by 40-50% from 50 sccm to 200 sccm, and clearly drops again at higher flow rates, to values at 600 sccm which are comparable to those obtained at 50 sccm. Moreover, operating at 9 Pa always yields higher etch rates compared to 5 Pa. This is expected because a higher pressure entails larger species densities in the reactor, which in turn results in a higher flux of F atoms to the wafer and hence a higher etch rate. The etch rate in this chemical system (i.e., SF<sub>6</sub> on Si) indeed only depends on the flux of F atoms (mainly) and on the flux of ions sputtering the wafer.

In this type of ICP reactor, the ICP source is quite remote from the wafer. As a result, a large fraction of ions that are created near the source will be lost through neutralization reactions before they arrive at the wafer. To check whether the etch rate is solely based on chemical etching by F atoms or whether the etch rate is indeed the result of both chemical etching and sputtering by ions, etch rate measurements were performed for different bias voltages.

As expected, a stronger bias voltage increases the etch rate, illustrating that sputtering by ions is also an important factor influencing the overall etch rate. Since sputtering is clearly important in this situation, it can be expected that not only SiF<sub>4</sub> will be an etch product, but also non-volatile SiF<sub>0.3</sub>. These non- (or low-)volatile species can redeposit on the reactor walls and on the wafer, hence

slightly reducing the etch rate. Therefore we would expect that the etch rate increases with gas flow rate. Indeed, a higher gas flow rate reduces the average time species spend in the reactor volume before being pumped out. If the non-volatile etch products have a longer residence time, the chance that they will redeposit on the wafer is higher, resulting in a lower etch rate. **Figure 3** shows the calculated gas residence times under the mentioned conditions. The different bias voltages do not have a significant effect on the residence time, unlike the gas flow rate and chamber pressure.

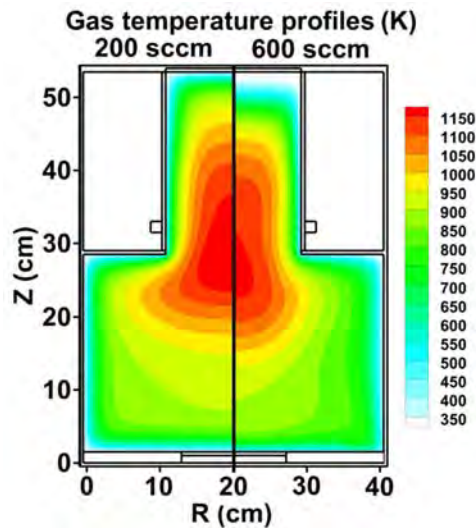


**Figure 3.** Calculated average residence time of the gas in the Alcatel 601E ICP reactor for 5 Pa and 9 Pa, as a function of gas flow rate in the range of 50-600 sccm.

From **figure 3** it is clear that the residence time decreases exponentially with gas flow rate, and thus the differences are most notable at lower gas flow rates. Assuming that the chance for redeposition on the wafer and the residence time are correlated, we can expect that the redeposition rate changes drastically at low flow rates, where redeposition might indeed play an important role. On the other hand, at high gas flow rates and thus short residence times, the sputtered species have a high chance of rapidly being pumped out, so we may expect that redeposition becomes less important. This chance of redeposition might thus explain why the etch rate initially increases with gas flow rate, but not why it drops again after 200 sccm, as was observed experimentally (see **figure 2**). Hence, we need to investigate other possible reasons.

During etching, the chamber pressure is regulated by the pump so that there is always a fixed pressure in the reactor. The plasma source, which is in this case the ICP coil, will heat up the gas during

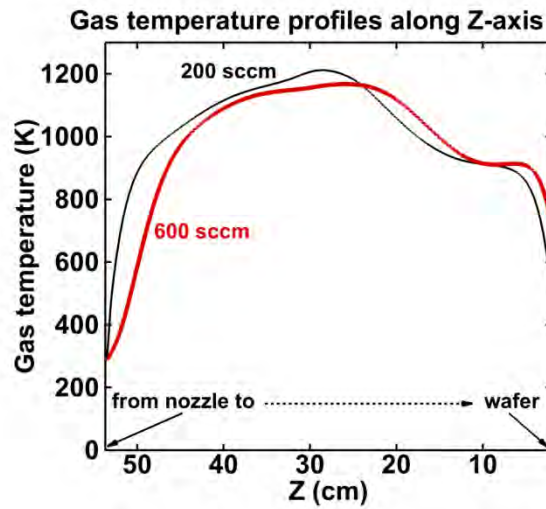
processing. When the gas temperature increases, the pump will lower the gas density to maintain a constant pressure. As a result, a higher gas temperature results in lower species densities and fluxes, and hence in a lower etch rate. This effect was indeed observed in our cryogenic experiments and simulations.<sup>17</sup> Following this logic, we would again expect a higher etch rate with higher gas flow rates, because the latter will more effectively cool the gas. In other words, at short residence times, the gas does not have enough time to be heated up by the plasma source. However, our calculations reveal that the reason for the drop in etch rate upon increasing gas flow rate after 200 sccm is because the gas temperature distribution within the reactor volume is altered as a function of gas flow rate. This is illustrated in **figure 4**, where the calculated two-dimensional gas temperature profiles are presented for 200 sccm and 600 sccm.



**Figure 4.** Calculated two-dimensional gas temperature profiles obtained for 200 sccm (left) and 600 sccm (right) gas flow rate, at 5 Pa and without bias.

The maximum temperature is found in the center of the tube near the position of the coil as expected, but the high temperature profile tends to be spread out more towards the wafer at 600 sccm compared to 200 sccm, as is clear from **figure 4**. The total average gas temperature at 600 sccm is lower due to the more efficient gas cooling at faster flow rates as mentioned earlier, but due to the higher vertical downwards laminar flow, the gas has less time to cool down before it arrives at the wafer. As a result, the gas tends to be slightly warmer near the wafer at 600 sccm compared to 200 sccm. For clarity, we

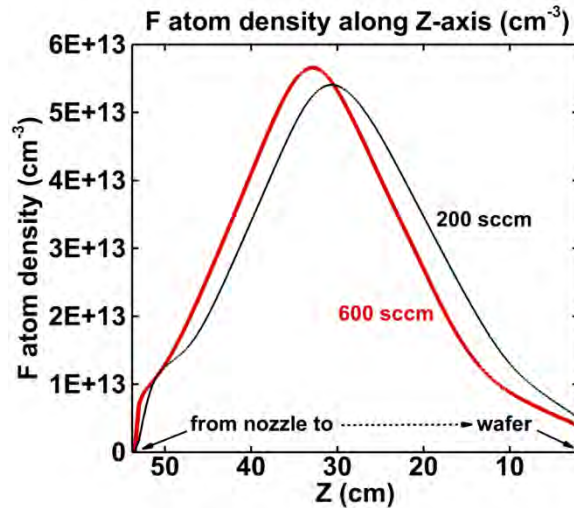
also present the gas temperature along the Z-axis from **figure 4**, in a X-Y plot, as illustrated in **figure 5**.



**Figure 5.** Calculated gas temperatures for 200 sccm (black line) and 600 sccm (red line) along the Z-axis, from the nozzle (at  $Z = 54$  cm) towards the wafer (at  $Z = 1.6$  cm), referring to the reactor geometry shown in figures 1 and 4.

In both cases (i.e., 200 sccm and 600 sccm), the gas is fed in the reactor at room temperature (i.e., 293 K). When the gas travels downwards through the tube, it heats up due to the plasma source (i.e., the coil at position  $Z = 32$ ). Due to the slower travel speed at 200 sccm, the gas has more time to heat up near the coil and therefore the gas temperature is higher in the upper area of the reactor at this flow rate. On the other hand, at 600 sccm, the gas travels faster and is thus heated less by the coil, but also has less time to cool down when travelling towards the wafer. Hence, in the lower part of the reactor and near the wafer, the gas temperature tends to be higher at 600 sccm compared to 200 sccm.

Due to the inverse correlation between F atom density and gas temperature, the calculated F atom density along the Z-axis line shows the opposite effect, as shown in **figure 6**.



**Figure 6.** Calculated F atom densities for 200 sccm (black line) and 600 sccm (red line) along the Z-axis travelling from the nozzle (at  $Z = 54$  cm) towards the wafer (at  $Z = 1.6$  cm) referring to the reactor geometry shown in figures 1 and 4.

At the nozzle, the  $\text{SF}_6$  gas is fed in the reactor and the F atom density tends to build up with a maximum near the coil, where most dissociation reactions take place due to the highest electron acceleration in this area. Due to wall losses and recombinations, the F atom density decreases when the gas travels from the coil towards the bottom of the reactor. It is clear from **figure 6** that the F atom density is slightly lower at 600 sccm near the wafer, due to the higher gas temperature in this area.

Because the F atom density in the plasma is directly correlated to the flux of F atoms towards the wafer, a lower etch rate is found at higher gas flow rates (i.e., above 200 sccm for this system). Indeed, the total (radially-averaged) F atom flux towards the wafer is calculated to be  $3.8 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$  at 600 sccm, while it is  $5.0 \times 10^{12} \text{ cm}^{-2} \text{ s}^{-1}$  at 200 sccm. It needs to be mentioned that the experimentally found drop in etch rates in the higher gas flow range (i.e., 40-50%) is more pronounced than the difference in F atom density and flux, predicted by the computational model (i.e., 20-25%), which can most probably be attributed to the fact that the size of the nozzle is limited to one computational cell size in the model, which is still larger than the size of the actual pin-like nozzle. As a result, the speed at which the gas is injected in the reactor is slightly underestimated in the model. However, we believe that the model still properly captures the correct effects to explain the experimentally observed trends.

Please note that F\* atoms, which are also very reactive towards Si, show a comparable density profile as the ground state F atoms as shown in **figure 6**, but with an overall density which is roughly 10000 times lower, so their influence on the overall etch rate is negligible.

It must also be noted that the electron density and electron temperature are unaffected by the change in gas flow rate. As a result, also the plasma potential does not change significantly as a function of gas flow. In other words, only the density profiles of the heavy particles (i.e., atoms, ions and molecules) are affected by the flow rate of the feed gas.

In this type of ICP reactor, the ICP source is quite remote from the wafer. As a result, a large fraction of ions that are created near the coil, will be lost through neutralization reactions before they arrive at the wafer. To check whether the etch rate is solely based on chemical etching by F atoms or whether the etch rate is indeed the result of both chemical etching and sputtering by ions, we decided to perform the experiments for different biases. It was found that the ions indeed have an important role in the etch process as it is clear that the etch rate increases significantly as a function of bias voltage. The total ion density follows a similar trend as the density of the F atoms, but is about factor of 100 lower overall.

It can thus be concluded that the etch rate is influenced by two simultaneous effects, which originate from tuning the total gas flow: (i) redeposition of non-volatile etch products, which entails a higher etch rate with higher gas flow rate, but becomes less important at higher flow rates, and (ii) downward shifting of the gas temperature, and thus a lower F atom density and flux near the wafer, which lowers the etch rate upon increasing gas flow rate, and which is less important at low gas flows. The combination of both effects results in the trend plotted in **figure 2** above, where the etch rate exhibits a clear maximum around 200 sccm in the range of 50-600 sccm.

From an economic point of view, it is advisable to keep the flow rate rather low and the pressure as high as possible, at least for this application. Indeed, increasing the gas flow rate, and hence the cost, by a factor of 4 (i.e., from 50 sccm to 200 sccm) will only yield an increase in etch rate of about 50-60% which is not sufficient to compensate for the higher gas cost.

For most anisotropic etch processes where ions play a major role in the trench or hole formation, operating pressures are usually not higher than 100 mtorr. It has been shown here that a higher

pressure yields a higher etch rate, but setting the pressure too high will entail undesired effects such as too much ion neutralizations before the ions arrive at the wafer and too much ion scattering when the sheath is no longer collisionless.

## **V. Conclusions**

We experimentally observed that the etch rate of Si with a SF<sub>6</sub> ICP is not linearly dependent on the total gas flow rate, but shows a maximum near 200 sccm in the range of 50-600 sccm. To explain this trend, we have numerically investigated the influence of the gas flow rate on the plasma characteristics.

We conclude that tuning the gas flow allows for control of two simultaneous effects that influence the etch rate, i.e., a higher gas flow rate results in (i) less redeposition of non-volatile etch products, which enhances the etch rate, and (ii) downward shifting of the temperature shape within the reactor volume, resulting in a higher temperature, and thus a lower F atom density and flux near the wafer, and hence a lower etch rate. The combination of both effects explains why the etch rate reaches a maximum at an intermediate gas flow rate of 200 sccm, in the range of 50-600 sccm. This study thus shows that finding the proper gas flow rate is very important in many situations for an optimized etch process in terms of etching rate.

From an economic point of view, it is desirable to keep the gas flow rate rather low, and the pressure as high as anisotropic etching allows. For this particular application, our results show that the etch rate only increases 50-60%, which is insufficient when the gas cost is increased by a factor of 4 (i.e., 50 sccm to 200 sccm).

## **Acknowledgements**

We are very grateful to Mark Kushner for providing the computational model. The Fund for Scientific Research Flanders (FWO; grant no. 0880.212.840) is acknowledged for financial support of this work. The work was carried out in part using the Turing HPC infrastructure at the CalcUA core facility of the Universiteit Antwerpen, a division of the Flemish Supercomputer Center VSC, funded by the Hercules Foundation, the Flemish Government (department EWI) and the University of Antwerp.

## References

- [1] Yamamoto A, Kawano Y, Nakai M, Nakagawa T, Sakugawa T, Hosseini H and Akiyama H, *IEEE Transactions on Plasma Science* **43** 3451 2015
- [2] Nakajima A, Uchida G, Kawasaki T, Koga K, Sarinont T, Amano T and Setsuhara, Y, *J. Appl. Phys.* **118** 043301 2015
- [3] Yambe K, Taka S and Ogura K, *Physics of Plasmas* **21** 043511 2014
- [4] Uchida G, Nakajima A, Takenaka K, Koga K, Shiratani M and Setsuhara Y, *IEEE Transactions on Plasma Science* **43** 4081 2015
- [5] Miao J, Yu D, Cao X, Xiang Y, Xiao M and Yao J, *Plasma Chem. and Plasma Proc.* **35** 879 2015
- [6] Li L, Leys C, Britun N, Snyders R and Nikiforov A, *IEEE Transactions on Plasma Science* **42** 2752 2014
- [7] Goto T, Inokuchi A, Ishibashi K, Yasuda S, Nakanishi, T, Kohno M and Ohmi T, *J. Vac. Sci. Technol. A* **27** 686 2009
- [8] Shi M and Clouet F, *J. Appl. Pol. Sci.* **46** 2063 1992
- [9] Monkowski J and Boyd K, *Solid State Technol.* **53** 20 2010
- [10] Lee J, Jung P, Devre M, Westermann R and Pearton S, *Solid State Electronics* **46** 685 2002
- [11] Ikegawa M, Kobayashi J and Fukuyama R, *J. Vac. Sci. Technol. A* **19** 460 2001
- [12] Ikegawa M, Ogawa Y, Fukuyama R, Usui T and Tanaka J, *J. Fluids Engineering* **124** 476 2002
- [13] Kwon B, Lee L, Lee N, Kim C and Choi C, *J. Korean Phys. Soc.* **62** 67 2013
- [14] Kwon B, Lee J and Lee N, *Thin Solid Films* **519** 6741 2011
- [15] Dussart, R, Boufnichel M, Marcos G, Lefauchaux P, Basillais A, Benoit R and Ranson P, *J. of Micromechanics and Microengineering* **14** 190 2003
- [16] d'Agostino R and Flamm D, *J. Appl. Phys.* **52** 162 1981
- [17] Tinck S, Tillocher T, Dussart R and Bogaerts A, *J. Phys. D: Appl. Phys.* **48** 155204 2015

- [18] Boufnichel M, Aachboun S, Lefauchaux P and Ranson P, *J. Vac. Sci. Technol. B* **21** 267 2003
- [19] Kushner M, *J. Phys. D: Appl. Phys.* **42** 194013 2009
- [20] Mao M, Wang YN and Bogaerts A, *J. Phys. D: Appl. Phys.* **44** 435202 2011