Energy distribution functions for ions from pulsed EUV-induced plasmas in low pressure N₂-diluted H₂ gas

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J. Beckers,^{1,a)} T. H. M. van de Ven,^{1,2} C. A. de Meijere,² R. M. van der Horst,² M. van Kampen,² and V. Y. Banine^{1,2}

AFFILIATIONS

¹Eindhoven University of Technology, Den Dolech 2, 5612 AZ Eindhoven, The Netherlands

²ASML, De Run 6501, 5504 DR Veldhoven, The Netherlands

^{a)}Author to whom correspondence should be addressed: j.beckers@tue.nl

ABSTRACT

The operation of Extreme Ultraviolet (EUV) lithography scanners inherently goes hand-in-hand with the creation of highly transient pulsed plasmas in the optical path of these tools. These so-called EUV-induced plasmas are created upon photoionization events when a pulsed beam of EUV photons travels through the low pressure background gas. It is fully recognized by the lithography industry that EUV-induced plasmas may significantly impact the quality and life-time of expensive and delicate optical elements in the scanner. Research efforts into EUV-induced plasmas impacting plasma-facing surfaces have so far been limited to pure hydrogen (H₂) plasmas. However, this hydrogen background gas may occasionally be diluted with a small fraction of another molecular gas such as nitrogen (N₂). The impact on the relevant plasma parameters of such molecular contaminants has remained unknown until now. In this letter, we put forward measurements of energy-resolved fluxes of (positive) hydrogen ions, nitrogen ions, and hydrogen-nitrogen ions created in a pulsed N₂-diluted EUV-induced plasma in H₂ at approximately 5 Pa (typical EUV lithography scanner conditions). The data have been obtained using an electrostatic quadrupole plasma analyzer and show that although the N₂-dilution fraction is small ($\sim 2 \times 10^{-3}$) compared to the H₂ partial pressure, implications for the ion flux out of the plasma and the composition thereof are significant. Since the mass of nitrogen-containing ions is much higher in comparison to that of their hydrogen counterparts, and because of their potential chemical activity, this effect has to be taken into account while studying the surface impact of EUV-induced plasmas.

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Ever since the introduction of Extreme Ultraviolet (EUV) lithography,¹ highly transient plasmas have been observed in the optical path of lithographic EUV scanners. These so-called EUV-induced plasmas are the peculiar result of the low pressure background gas being irradiated with high energy photons with wavelengths in the EUV range (~ 13.5 nm).² Although these plasmas are both highly transient and rarified, the created environments satisfy the three “formal criteria” in order to be called a plasma.³ EUV-induced plasmas are recognized as an inevitable phenomenon impacting the quality, the life-time, and the long-term stability of the EUV optical components used. For instance, impacting species generated by EUV-induced plasmas may damage multilayer structures,^{4–7} while under the right conditions, the same plasma can be utilized for *in-situ* cleaning purposes.^{8–10}

The density and the dynamics of one of the dominant plasma species in these plasmas, i.e., the electrons, have been investigated numerically^{11,12} and experimentally by means of Langmuir probes¹³

and temporally resolved Microwave Cavity Resonance Spectroscopy (MCRS) in low pressure argon^{14–16} and hydrogen^{16,17} environments. Double-mode MCRS¹⁸ and multi-mode MCRS¹⁹ have revealed this information on spatially resolved, while the combination of MCRS measurements and an ambipolar diffusion model showed that in the moderately high pressure regime (>3 Pa in argon²⁰ and >6 Pa in hydrogen³), electrons thermalize to values close to room temperature in the temporal afterglow of these pulsed plasmas.²⁰ Especially of interest when studying the interaction of EUV-induced plasmas with plasma-facing surfaces are the total ion flux and the ion energy distribution functions (IEDFs) of the ionic compounds impinging upon the surface. In the literature, two diagnostics have previously been used to study ion fluxes from EUV-induced plasmas; a Retarding Field Energy Analyzer (RFEA) and an Electrostatic Quadrupole Plasma analyzer (EQP).^{21,22} So far, these studies have been limited to EUV-induced plasmas in pure hydrogen (H₂). However, the addition of even small

amounts of other molecular impurities [e.g., nitrogen (N_2)] at the level of 0.1%–1% can become highly relevant for the ionic balance in the system and for the possible impact on plasma-facing surfaces.

In this letter, we report on measurements of IEDFs for 10 specific ions in admixtures of H_2 and ($\sim 0.2\%$) N_2 .

For the present study, we used a configuration similar to the one used during our previous experiments on EUV-induced plasmas in undiluted H_2 gas.²¹ While the reader is referred to Refs. 21 and 23 for extensive details, here, we discuss only the main components of the setup which are depicted in Fig. 1. Three vacuum chambers were connected. The source chamber housed a xenon-based discharge produced plasma (DPP) source generating pulsed EUV radiation with a repetition rate of 500 Hz and a pulse duration of 50 ns [full-width-at-half-maximum (FWHM) value]. Eight rotationally symmetric hyperboloid and ellipsoid collector mirrors of grazing incidence type in the collector chamber focused this radiation into the measurement chamber. At the focal point, the diameter of the EUV beam was 4 mm, while its divergence was 10° . At the location where the measurements were performed, the beam diameter was 32 mm, and an EUV pulse energy of $114 \pm 6 \mu J$ was measured. This resulted in an EUV intensity at the position of the front cap of the EQP of $\sim 283 \text{ W/cm}^2$, which is comparable with conditions employed in lithography tools.

Just before the radiation entered the measurement chamber, it passed through an optical filter. This filter was a Si:Zr membrane of 50 nm thickness and was used to filter out the out-of-band ($>20 \text{ nm}$) radiation. The source's radiation spectrum was previously measured by Kieft,²⁴ while several other researchers convoluted this spectrum with the transmission curve of the used optical filter in order to retrieve the spectrum at the position where the measurements were performed.^{3,23} Before and after each experiment, the stability of the spectrum-integrated EUV pulse energy was confirmed using a calorimetric power sensor as introduced and described by Van der Horst *et al.*¹⁵

The focused EUV radiation interacted with the injected gas around the axis of a cylindrical measurement volume (denoted "Cylinder" in Fig. 1) made of aluminum and having an inner diameter and an inner height of both 100 mm. The reason for having the experimental geometry limited by this cylinder was twofold. First, it aids the interpretation of the results, e.g., for future comparison with MCRS measurements. Second, it facilitates numerical modeling efforts by providing a well-defined volume. The front-end of the cylinder

contained an aperture which allowed the EUV beam to enter the cylinder. The back-end was closed by an aluminum plate that accommodated the front-cap of the EQP. The side-wall of this cylinder contained two additional slits; one for sliding in the calorimetric EUV power sensor in the beam path and the other for future experiments where the field of view of the EQP is perpendicular to the EUV beam (not used in this work). All walls of the measurement cylinder were electrically grounded.

Since the openings in the walls of the cylinder were significant, it can safely be assumed that the composition and the pressure of the gas in the cylinder were the same as set in the enclosing measurement chamber ($\sim 5 \text{ Pa}$ working pressure and $\sim 10^{-5} \text{ Pa}$ base pressure). Differential pumping ensured that the gas type and the working pressure inside the measurement chamber could be controlled independent of the gas type and the working pressure in the source chamber and in the collector chamber. Partial pressures of H_2 and N_2 were controlled by mass flow controllers and verified with the EQP in the residual gas analyzer mode.

The needed positive ion diagnostics were enabled by an EQP1000, Hidden Analytic Ltd. This device is able to measure the energy, the mass, and the time-resolved ion fluxes towards the surface of its front cap. For a fully detailed description, the calibration, and the alignment procedure, the reader is referred to Ref. 23. Here, we only discuss the basic specifications of the EQP used. Ions were sampled through an orifice after which they were transported through a flight-tube which extended 400 mm into the measurement vessel. The collected ions are the energy and the mass filtered before being detected by the secondary electron multiplier (SEM) ion detector at the end. The diameter of the sampling orifice of $20 \mu m$ was chosen such that it was sufficiently small (compared to the expected Debye length, e.g., $\sim 43 \mu m$ for an electron temperature (1 eV) and an electron density ($3 \times 10^{16} \text{ m}^{-3}$) that are typical for these kinds of plasma²¹) to prevent the generated plasma to enter the flight tube. The used SEM had a large dynamic range of 7 orders of magnitude, and the mass filter was set to an ion mass range of 1–50 amu. Before being detected, the trajectories of the incoming ions were "steered" by 15 individually adjustable electrostatic lenses. The procedure for setting these lenses and the calibration of the full EQP is extensively described in Ref. 23.

Time-averaged IEDFs for specific positive hydrogen ions (H^+ , H_2^+ , and H_3^+), positive nitrogen ions (N^+ , N_2^+ , and N^{2+}) and positive nitrogen-hydrogen ions (NH_2^+ , NH_3^+ , NH^+ , and N_2H^+) have been obtained from an EUV-induced plasma in a gas admixture of 5 Pa H_2 and 0.01 Pa N_2 . The results are plotted in Figs. 2(b), 3(a), and 3(b), respectively. As reference, the IEDFs for positive hydrogen ions from an EUV-induced plasma in undiluted H_2 (Ref. 21) are shown in Fig. 2(a).

As can be seen from Fig. 2(b), when comparing with the pure H_2 situation in Fig. 2(a),²¹ diluting the H_2 gas with a small fraction of N_2 hardly alters the time-averaged IEDFs for the hydrogen ions; H_3^+ remains the dominant (positive hydrogen) ion and the IEDF for H^+ continues to show the energetic tail that extends to almost 20 eV. However, even though the partial pressure ratio between N_2 and H_2 is low ($2 \cdot 10^{-3}$), detected fluxes of nitrogen ions are significantly large compared to those of hydrogen ions (e.g., a ratio of $\sim 2 \cdot 10^{-2}$ when comparing, for instance, the maximum IEDF values for N_2^+ and H_3^+). The reason for this is that the total photoionization cross-section at 13.5 nm for N_2 ($\sigma_{pi} = 2.5 \cdot 10^{-22} \text{ m}^2$, Ref. 25) is more than

FIG. 1. Schematic overview of the experimental setup used (not to scale) with the source chamber housing the xenon-based discharge produced plasma source, the collector chamber housing the focusing optics, and the optical filter and the measurement chamber housing the measurement cylinder and the EQP diagnostics. Reproduced with permission from Van De Ven *et al.*, J. Appl. Phys. **123**, 063301 (2018). Copyright 2018 AIP Publishing.

FIG. 2. Time-averaged IEDFs for H^+ , H_2^+ , and H_3^+ ions from an EUV-induced plasma produced (a) in undiluted H_2 at 5 Pa (used data from Ref. 21) and (b) in H_2 at 5 Pa diluted with 0.01 Pa N_2 (this work).

40 times larger than the corresponding value for H_2 ($\sigma_{pi} = 6 \cdot 10^{-24} \text{ m}^2$, Refs. 26 and 27). At this pressure, electron impact ionization is of significant importance when it comes to continued creation of plasma species even after the EUV pulse has ended [typical after 50 ns–100 ns (FWHM) after the start of the pulse].¹⁷ Immediately after the first photoionization events, the energy of the created electrons (energy-distributed highly non-Maxwellian) equals the energy difference between the photon energy and the ionization energy of the respective molecules. At these energies, however, the cross-section σ_{ei} for electron impact ionization is only slightly higher for N_2 ($\sigma_{ei} = 1.5 \cdot 10^{-20} \text{ m}^2$ for N_2 , Ref. 28 compared to $\sigma_{ei} = 1.1 \cdot 10^{-20} \text{ m}^2$ for H_2 , Ref. 29). Therefore, the relatively high value of the flux of nitrogen ions can be contributed mainly to the much higher photoionization cross-section (in the EUV range) of N_2 compared to that of H_2 .

With respect to the nitrogen ions in Fig. 3(a), the first surprising feature is the energetic tail in the IEDF for N^+ , while the IEDFs for the other ions do not show this feature. Similar observations can be made from Fig. 2 (and have been made before²¹) for the hydrogen ions; the IEDF for H^+ shows an energetic tail, while the IEDFs for the other hydrogen ions do not. Several mechanisms can be involved in the

FIG. 3. Time-averaged IEDFs from an EUV-induced plasma produced in H_2 at 5 Pa diluted with 0.01 Pa N_2 . Detected ions are plotted in groups regarding the contained elements. (a) nitrogen ions: N^+ , N_2^+ , and N_2^{2+} and (b) nitrogen-hydrogen ions: NH_2^+ , NH_3^+ , NH^+ , and N_2H^+ .

production of various ions. In a direct sense, photoionization [Eq. (1)] and double photoionization [Eq. (2)] of N_2 can occur upon EUV irradiation

The reaction product of the reaction in Eq. (1) N_2^+ is, for low ion energy values, the dominant nitrogen ion. However, N_2^+ may be subject to electron impact dissociation: $\text{N}_2^+ + e^- \rightarrow \text{N}^+ + \text{N} + e^-$ with a threshold of $8.4 \pm 0.5 \text{ eV}$ and a kinetic energy release of $6.4 \pm 0.5 \text{ eV}$.³⁰ Although double photoionization [reaction in Eq. (2)] might be able to produce doubly ionized nitrogen molecules (N_2^{2+}), these molecules have not been detected by the EQP measurements. From that, it can be concluded that in this case, N_2^{2+} is unstable and dissociates before being detected. Iwayama *et al.* experimentally showed the possible dissociation channels for N_2^{2+} to be either $\text{N}_2^{2+} \rightarrow \text{N}^+ + \text{N}^+$ or $\text{N}_2^{2+} \rightarrow \text{N}^{2+} + \text{N}$.³¹ Note that both the ionic reaction products N^+ and N^{2+} are detected [see Fig. 3(a)]. Pandey *et al.*³² showed that

dissociation of high-lying repulsive N_2^{2+} states into $2N^+$ governs a kinetic energy release between 10.7 eV and 14.8 eV. Distributing this energy over the two reaction products and superimposing this on the directed “bulk” energy the ions pick up from the plasma-created space charge region (e.g., the shape of the IEDF for N_2^+) perfectly explain the presence, the absolute value and the shape of the high-energy tail in the IEDF for N^+ . Note that—after being accelerated by the electric fields in the space charge region—the bulk energy of N^{2+} is expected to be twice the bulk energy of N^+ . This, however, is not observed in the respective IEDFs, which can be explained by the fact that N^{2+} is also accelerated twice as fast by the electrostatic fields in the EQP’s energy filter. As a result, they are detected at half the pass energy of the filter.

Besides these photoionization events, initially created fast electrons might be responsible for the further production of ionic species through electron impact ionization processes.

From Fig. 3(b), it can be observed that a variety of plasma chemical reactions produce significant amounts of (positive) nitrogen-hydrogen ions, i.e., NH_2^+ , NH_3^+ , NH^+ , and N_2H^+ ions. Note that NH_4^+ , an ion often found in plasmas in H_2/N_2 mixtures, is not detected at all. Apparently, plasma chemical pathways towards NH_4^+ are unlikely under these conditions. Among the detected nitrogen-hydrogen ions, the most dominant one is N_2H^+ . This ion is most likely created by the proton-transfer reaction between H_3^+ and N_2

As observed experimentally before²¹ and also from the measurements presented in Fig. 2(b), H_3^+ is highly abundant. The reason for the abundance of H_3^+ —and therefore also for the importance of this species in the plasma chemical reaction in Eq. (3)—is the highly efficient reaction $H_2^+ + H_2 \rightarrow H_3^+ + H$. This “proton-hop-reaction” is governed by a very large cross-section³³ since H_2^+ induces a dipole moment in the neutral H_2 molecule, creating an attracting potential between the two species.

Finally, it can be observed that the IEDFs for N_2^+ and N_2H^+ show a cut-off at an energy (around 5 eV) that is much lower than the maximum energies observed for all other ions (>7.5 eV). From the current experiments, this effect is not understood yet. However, it might be correlated with the higher mass-over-charge-ratio of these ions; N_2^+ and N_2H^+ are much heavier than the ions housing a single N atom. As a result, the transit time through the plasma-induced space charge region,^{11,17,21} accelerating the ions towards the front-cap of the EQP, becomes longer. At the same time, this space charge region is highly transient. Due to electron cooling, the accelerating electric fields may have been depleted before the ions even have transited the full initial space charge region. Hence, these heavier ions may not “feel” the full potential difference over the initially plasma-induced space charge region.

Overall, the presented measurements in this letter are for EUV lithography scanner realistic conditions in terms of the gas composition, i.e., N_2 -diluted H_2 . The main conclusion is that although the fraction of N_2 additives is very small, a considerable amount of nitrogen (containing) ions is produced. This fact is crucial for the lithography industry to take into account because of the higher mass (and thus a higher impact) of these additional ions and because of their potential chemical activity regarding the exposed surfaces.

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