

The evolution of a hydrogen-depleted surface layer on a-C:H during bombardment with N₂⁺ ions.

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1. Introduction

Nitrogen seeding is presently used in fusion devices to reduce the local power load on highly exposed surfaces by enhanced radiative cooling [1]. This is why the interaction of nitrogen ions with amorphous hydrogenated carbon (a-C:H) thin films, which are the result of co-deposition of carbon and hydrogen isotopes in carbon containing fusion devices, is of considerable interest [2, 3]. During the interaction of energetic ions with hydrocarbon surfaces a modified surface layer builds up with different properties compared to the bulk film [4]. Bond breaking within the ion penetration depth modifies the carbon network and leads to a hydrogen-depleted layer. In steady state the top layer is dynamically reformed and its underlying bulk becomes thinner. Up to now key parameters like the sputtering yield could only be detected for that steady state erosion but were not accessible for the transient phase. Here we report on the first experimental measurements of the sputtering yield during this transient phase.

2. Experiment

In our experiments a highly accurate quartz crystal microbalance (QCM) developed at Vienna University of Technology [5, 6] was utilized to monitor the mass change of plasma deposited a-C:H thin films in-situ and in real-time during the bombardment with N₂⁺ ions at 460 K for various energies. To this purpose, soft polymerlike a-C:H layers with a hydrogen content of 50% were deposited onto one of the polished gold electrodes of several identical quartz crystals in an ECR methane plasma [7]. The film growth and characteristics was monitored with in situ ellipsometry. The thickness of the films was determined to be about 370 nm. The mass sensitive part of our QCM is a stress compensated (SC) cut quartz crystal driven at its thickness shear mode at a resonance frequency of approximately 6 MHz by a highly accurate electronics built in Vienna [5]. Transfer to the irradiation experiment

MAJESTIX (for a detailed description see [8]) took place in air. Molecular N_2^+ ions were extracted from the filament ion source by a voltage of 1000 V, passed through a Wien-filter and subsequently decelerated to a final impact energy of 150 eV and 250 eV, respectively (fig. 1). For each ion impact energy a freshly prepared a-C:H film was used. The ion optics in the beam-line allows to create a homogenous irradiated area of approximately 10 mm in diameter. Measurements were performed for a target temperature of 460 K, because at this temperature the quartz frequency is least sensitive to small temperature changes [5], which could be induced by power deposition of the impinging ions.

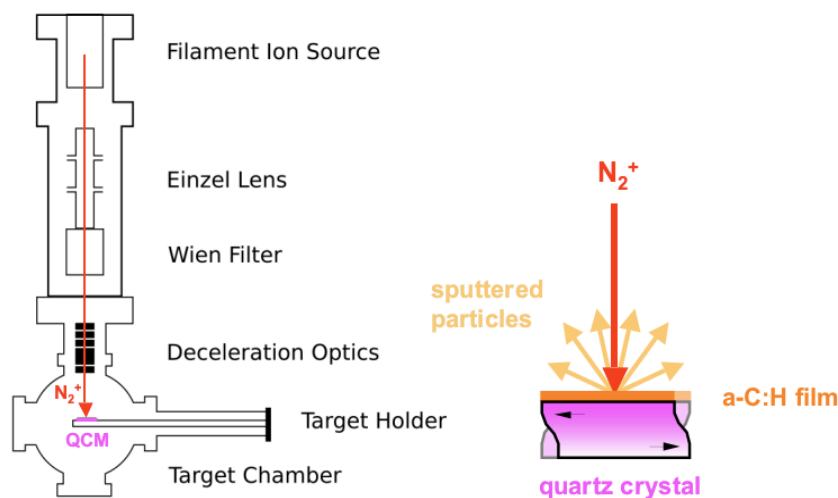


Fig.1: Experimental setup MAJESTIX [8] at IPP Garching (left). Schematic illustration of the QCM (right).

According to Sauerbrey [9] the total mass change Δm of a thin layer deposited onto one of the electrodes of the quartz can be deduced from the change in the resonance frequency f of the quartz crystal.

$$\frac{\Delta f}{f} = -\frac{\Delta m}{m} \quad (1)$$

Small frequency drifts of the QCM are usually corrected by observing the QCM frequency during “beam off” phases (cf. [6]). To accurately determine the total erosion/sputtering yield, which in our experiment is measured in atomic mass units (amu) per incident (molecular) ion, the impinging ion current density on the active area of the crystal has to be known and kept constant as well as homogenous throughout the whole experiment. The ion beam profile was quantitatively measured and monitored by a Faraday cup located on the sample holder. Mass changes down to 10^{-5} $\mu\text{g/s}$ can be detected [5, 6]. This corresponds to a removal of 10^{-2} a-C:H monolayers per second.

3. Results and discussion

Fig. 2 shows, how the QCM frequency measured for impact of 250 eV N_2^+ ions changes its slope with increasing ion dose, indicating a significant change in erosion yield during the course of the measurements. Corresponding sputtering yields as a function of the ion dose are shown in fig. 3 for both 150 eV and 250 eV N_2^+ ion impact energy. Measurements were always started with a fresh plasma deposited a-C:H layer. At the beginning of the ion irradiation an elevated sputtering yield is observed that decreases exponentially with fluence to approximately $1/3$ of its initial value. The steady-state values reached after a fluence of typ. 10^{15} ions/cm² agree well with sputtering yields observed for hard (30% H content) a-C:H films determined at room temperature [2, 3] (see fig. 4) and which were explained by a considerable contribution of chemical sputtering by N_2^+ at these low kinetic energies.

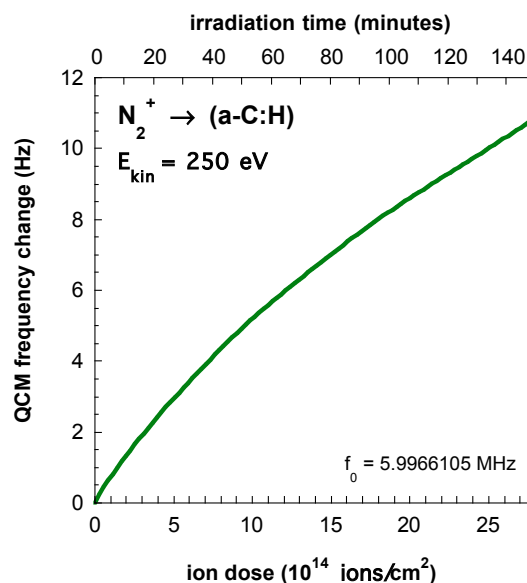


Fig. 2 Evolution of the quartz frequency during 250 eV N_2^+ ion bombardment of soft a-C:H as a function of irradiation time and ion dose.

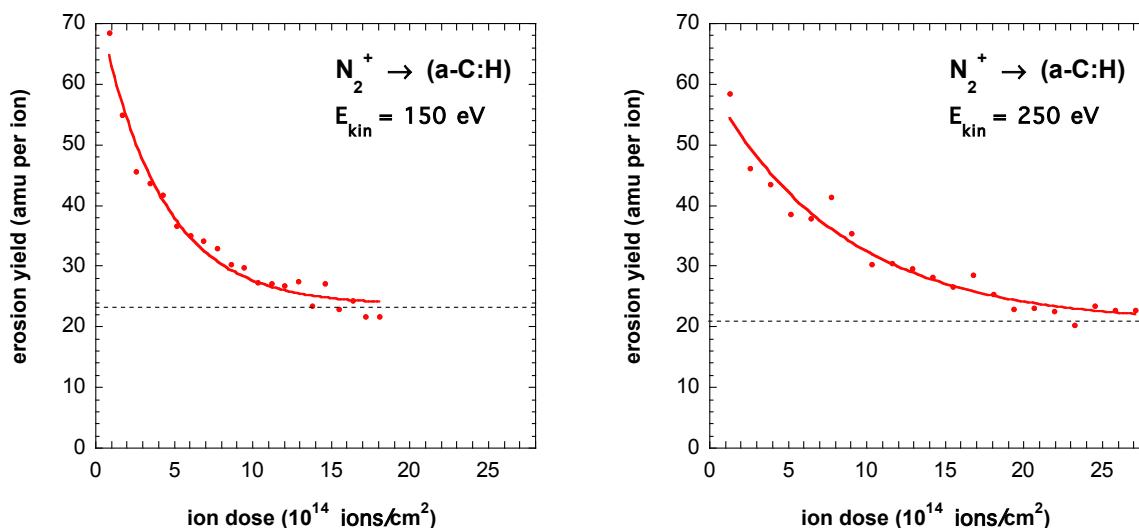


Fig. 3: The total erosion yield (in atomic mass units per incident N_2^+ ion) as a function of ion dose was evaluated from the slope of the frequency curve shown in fig. 2.

Mind, that from our method the total mass change (i.e. including H and N atoms) per incident ion is derived [5], while the technique used in [2] determines the number of eroded carbon atoms. From fig. 3 it can also be seen that the steady state erosion yield for 250 eV

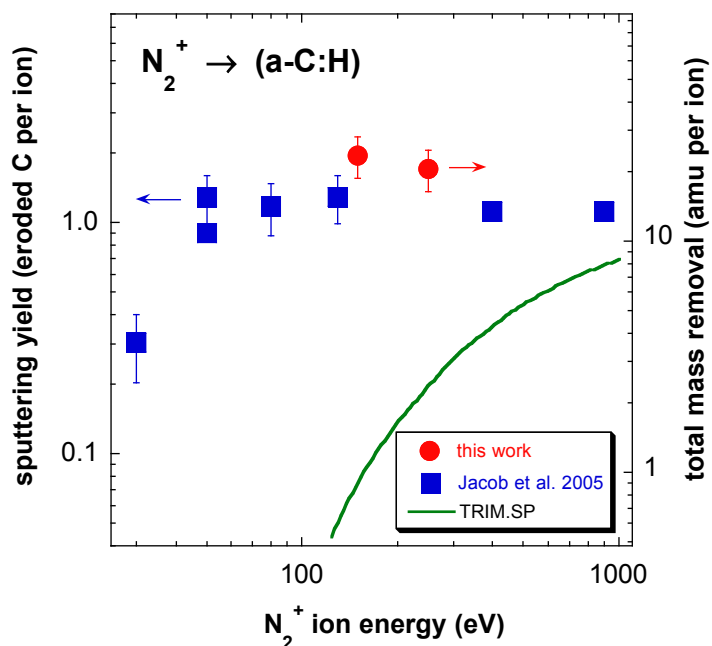


Fig. 4: Comparison of the obtained steady-state sputtering yields for impact of N_2^+ ions on soft a-C:H with previous data [3] for hard a-C:H and the results of TRIM.SP calculations [3, 11], which only show kinetic sputtering contributions.

impact energy ions is reached at higher fluences than for projectiles of 150 eV kinetic energy. We have estimated nitrogen projectile ranges in a-C:H using SRIM2008 [10]. Depending on the hydrogen content of the layer an implantation depth of 8-12Å is calculated for a 75 eV N atom (corresponding to a 150 eV N_2 molecule) as compared to 10-15Å for a 125 eV N atom (250 eV N_2). The fluences where saturation is observed have the same scaling with respect to

projectile energy as the implantation depths calculated by SRIM.

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References

- [1] J. Rapp et al., *J. Nucl. Mat.* **337–339**, 826 (2005).
- [2] W. Jacob, C. Hopf and M. Schlüter, *Appl. Phys. Lett.* **86**, 204103 (2005).
- [3] M. Schlüter, C. Hopf and W. Jacob, *New J. Phys.* **10**, 053037 (2008).
- [4] A. von Keudell, W. Jacob and W. Fukarek, *Appl. Phys. Lett.* **66**, 1322 (1995).
- [5] G. Hayderer, M. Schmid, P. Varga, HP. Winter and F. Aumayr, *Rev. Sci. Instrum.* **70**, 3696 (1999).
- [6] A. Golczewski, K. Dobes, G. Wachter, M. Schmid and F. Aumayr, *Nucl. Instr. Meth. B* **267**, 695 (2009).
- [7] T. Schwarz-Selinger, A. von Keudell and W. Jacob, *J. Appl. Phys.* **86**, 3988 (1999).
- [8] W. Jacob, et al., *Rev. Sci. Instrum.* **74**, 5123 (2003).
- [9] G. Sauerbrey, *Z. Phys.* **155**, 206 (1959).
- [10] SRIM computer code, available at <http://www.srim.org>
- [11] W. Eckstein, *Computer Simulation of Ion-Solid Interactions*, Springer Series in Materials Science, 1st ed. Springer, Berlin, (1991).