



Transient effects during sputtering of a-C:H surfaces by nitrogen ions

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ABSTRACT

Sputtering of polymer-like amorphous hydrogenated carbon (a-C:H) thin films by 0.5–1 keV N_2^+ molecular ions has been studied in situ and real-time using a highly sensitive quartz crystal microbalance technique. During bombardment of a fresh, plasma-deposited a-C:H layer with nitrogen ions the measured sputtering yield decreases exponentially with ion fluence until a steady state value is reached at a fluence of typically about $3.5 \times 10^{15} N_2^+$ ions per cm^2 . A chemical sputtering mechanism has to be considered in addition to physical sputtering to explain the observed steady state sputtering values. Simulations based on the code TRIDYN, which take into account a change of surface composition due to implantation and erosion, are performed to understand the transient development of sputtering yields.

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1. Introduction

In present fusion experiments carbon is still often used as first wall material because of its advantageous thermal properties. Carbon as a plasma-facing component, however, poses a serious problem for future fusion devices. Co-deposition of eroded carbon with hydrogen isotopes leads to formation of hydrogenated amorphous carbon (a-C:H) layers which contain tritium. As a consequence a tritium inventory within the fusion vessel builds up over time and under reactor-like conditions is expected to rapidly exceed required safety limits [1].

Nitrogen seeding is presently used in fusion devices to reduce the local power load on highly exposed surfaces by enhanced radiative cooling [2]. This is why the interaction of nitrogen ions with a-C:H thin films is of considerable interest. It has recently been shown [3,4] that a-C:H layers bombarded by molecular nitrogen N_2^+ ions exhibit an erosion rate which is enhanced by the chemical reactivity of the nitrogen projectiles especially at ion energies below the threshold for physical sputtering. By this, apart from radiatively cooling the edge plasma, the use of nitrogen as a seeding gas could also have a positive effect on the tritium inventory by eroding re-deposited a-C:T layers within a fusion device.

Hopf et al. [5] were able to model the energy dependence of this so-called chemical sputtering process on the basis of the following microscopic picture: when penetrating the a-C:H layer, energetic ions break C–C bonds, which are eventually passivated by

consecutive projectiles. In the case of chemically reactive projectiles, this leads to the formation of volatile species, which can diffuse to the surface and desorb there. Hopf et al. [5] showed that the chemical sputtering yield is thus proportional to the bond breaking density, i.e. the number of carbon displacement events per depth interval $y_{bb}(x,E)$, and the ion implantation depth profile $n(x)$. According to the above mechanism chemical sputtering requires the presence of chemically reactive species such as hydrogen or (in our case) nitrogen, while physical sputtering does not. Since chemical sputtering only takes place at sites where reactive atoms (i.e. hydrogen or nitrogen) are present, the corresponding yield is thus assumed to be proportional to their respective atomic fractions within the target, while physical sputtering is proportional to the complementary fraction, i.e. the number of ‘pure’ carbon sites, where no reactive species is available in the vicinity. By this the authors in [5] were furthermore able to describe the dependence of the sputtering yield on the incoming ion flux.

In this work we report on investigations of the total sputtering yield of polymer-like, ‘soft’ a-C:H layers with a hydrogen content of 50% under N_2^+ ion bombardment using a highly sensitive quartz crystal microbalance technique (Section 2). Due to this high hydrogen content, ‘soft’ a-C:H layers exhibit the characteristics of an insulating layer, while ‘hard’ a-C:H films contain less hydrogen (typically 30%) and are better conducting surfaces. When exposing a fresh a-C:H layer to a nitrogen ion beam we find, that the erosion yield at first exponentially decreases with ion fluence until a steady state value is reached after applying some $10^{15} N_2^+$ atoms to the surface (Section 3). We use the above-described chemical sputtering model by Hopf et al. [5] to explain our measured steady state

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sputtering yields and present simulation calculations based on the code TRIDYN to understand the transient development of sputtering yields (Section 4).

2. Experimental setup

Total sputtering yields of ‘soft’ a-C:H layers under nitrogen bombardment are investigated at TU Wien under controlled laboratory conditions with a self-built quartz crystal microbalance (QCM) setup (Fig. 1). The mass sensitive part of the QCM is a stress compensated (SC) cut quartz crystal driven at its thickness shear mode with a resonance frequency of about 6 MHz. The QCM is operated at a temperature of 460 K, where temperature changes induced by power deposition of the impinging ions have a minimal effect on the frequency. Sophisticated electronics permits the detection of mass changes of as small as 10^{-2} a-C:H monolayers per second. A detailed description of the QCM setup can be found elsewhere [6–8].

In an ECR methane plasma discharge a ‘soft’, polymer-like a-C:H layer with a hydrogen content of 50% is deposited onto one of the polished gold electrodes of the quartz crystal [9]. The film growth and characteristics of the layer was monitored by in situ ellipsometry. The thickness of the films was determined to be about 370 nm. The sample is then transferred ex vacuo to an UHV experimental chamber, which is connected to the ECR ion source facility at Vienna University of Technology. It is mounted onto a sample holder, which is equipped with a Faraday-cup for ion current determination and which is furthermore rotatable and moveable in xyz direction.

Molecular nitrogen ions are produced in a 14.5 GHz ECR ion source [10]. After extraction they are mass-over-charge selected with a sector magnet and focused onto the sample holder by means of an electrostatic lens system (Fig. 1). A pair of deflection plates right in front of the target is used to uniformly irradiate the active area of the quartz crystal by rapidly scanning the ion beam over the surface. By this, the mechanical stress imposed onto the quartz by a non-uniform erosion of the target film can be minimized and an accurate determination of the effective ion current density is possible. The Faraday-cup is used to scan the beam profile and monitor the ion current in between individual measurements.

The total mass change of the target film is determined from the Eigen-frequency change of the quartz crystal $\Delta f/f = -\Delta m/m$ [11]. From this the absolute mass loss per incident ion is determined by using the relation

$$Y = \frac{(\rho_Q l_Q)/(f) \Delta f}{I/(e_0 A) \Delta t} \quad (1)$$

where ρ_Q is the quartz’ density, l_Q its thickness and f its resonance frequency and $I/(e_0 A)$ is the ion current density per unit area impinging on the quartz crystal.

In order to accurately determine the mass change under ion irradiation, the frequency change of the quartz crystal induced by the N_2^+ beam is corrected by the quartz crystal’s intrinsic frequency drift at rest. For this purpose, measurements with ion beam are periodically alternated by ‘beam-off’ periods (for details see [6]). The sputtering yield, which in our experiment is measured in atomic mass units (amu) per incident projectile, is derived from the slope of a fit to the frequency change-curve. To investigate the evolution of a non-constant sputtering yield, consecutive measurements can be re-joined to a single curve. We note, that the QCM technique is only able to detect total mass changes, i.e. “net erosion” or “net deposition”, but does not deliver information on actually sputtered species.

3. Experimental results

Fig. 2 shows, how the QCM frequency measured for impact of 1 keV N_2^+ ions (i.e. 500 eV per N-atom) changes its slope with increasing ion fluence, indicating a significant change in erosion yield during the course of the measurements. The corresponding evolution of the sputtering yield as a function of the ion fluence is shown in Fig. 3. The measurements started with a virgin, i.e. freshly deposited ‘soft’ a-C:H layer. Fluences and sputtering yields are given per incident nitrogen atom (not N_2 molecule). At the beginning of the ion irradiation an elevated sputtering yield is observed that decreases exponentially with fluence to approximately 1/5 of its initial value. After approximately 7×10^{15} N atoms per cm^2 have been applied to the surface a steady state value of the sputtering yield is finally reached (Fig. 3). At this fluence approximately 4 nm of target material have been removed, as can be estimated from the observed sputtering yield and with a target density of 1.1 g/cm^3 [3]. A similar behavior was also found for other impact energies (500 eV and 700 eV N_2^+ projectiles), always using a fresh a-C:H film of course.

Fig. 4 summarizes the steady state sputtering yields obtained at the end of each measurement series. For comparison data by Jacob et al. [3] for bombardment of ‘hard’ a-C:H layers are shown. These data were obtained for a-C:H layers of lower hydrogen content (30%), than in our case. Mind also, that in [3] the number of eroded carbon atoms per incident N-atom is determined as a direct consequence of the measurement technique. With our technique on the other hand, the total mass removal (including hydrogen, nitrogen and heavier compounds such as different kind of hydrocarbons C_xH_y and other volatile molecules) is obtained. Both differences might explain the deviations between our data and those of Ref. [3].

The code TRIDYN [12] has been used to perform simulations of the interaction of nitrogen with a-C:H (see also Section 4). In these simulations the molecular nitrogen ion is treated as two separate atomic nitrogen projectiles at the same velocity or equivalently at the same specific energy (i.e. half the impact energy). When just

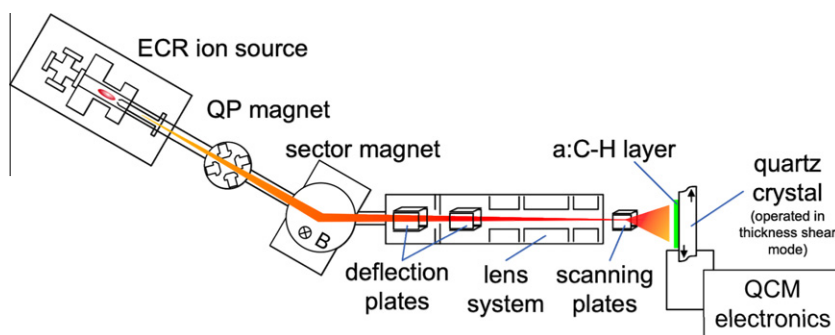


Fig. 1. Schematics of the experimental setup (not to scale).

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