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# Interaction between seeding gas ions and nitrogen saturated tungsten surfaces

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ABSTRACT

In a fusion reactor, where nitrogen is used as a seeding gas to mitigate the heat load to the plasma facing components, it is expected that mixed tungsten-nitride surfaces will build-up during operation. For fusion research it is of major concern to investigate the influence of such mixed material layers on physical properties of the plasma facing components such as e.g. their sputtering yield, their electrical conductivity or their melting point. Sputtering of pure tungsten W, and nitrogen saturated tungsten W-N surfaces under the impact of argon $Ar^{q+}$ ($q = 1–9$) and neon ions $Ne^{q+}$ ($q = 1, 4$) was therefore investigated under controlled laboratory conditions using a quartz crystal microbalance technique. Total sputtering yields were determined in an impact energy range of 100–5000 eV. Particular emphasis was put on testing any charge state dependency of the sputtering yield (i.e. potential sputtering), which was not found for neither of the investigated surfaces.

## 1. Introduction

Carbon is increasingly being replaced by metallic plasma facing surfaces in fusion machines. On the one hand, its chemical reactivity with hydrogen isotopes results in unacceptably high erosion rates due to chemical sputtering, which ultimately limits the lifetime of carbon containing materials in a fusion reactor vessel. On the other hand the formation of hydrogenated carbon layers by co-deposition of carbon impurities with hydrogen isotopes throughout the fusion machine would drive the tritium inventory in the vacuum vessel beyond operation limits within only a comparably small number of discharges [1].

The transition to all-metal machines however, involves dismissing the benign cooling effect of radiating carbon species in the plasma edge. To mitigate the heat loads to plasma facing components by radiative power dissipation, it will therefore be necessary to replace intrinsic carbon impurities by the injection of seeding gases. Potential seeding species involve noble gases like argon or neon, but also nitrogen, which has proven to be a valuable substitute for carbon in the divertor region [2]. In ASDEX Upgrade nitrogen seeding with power load feedback has already matured into a standard operational scenario [2].

The erosion of high Z plasma facing components will be dominated by these externally seeded impurity species. Moreover, implantation of nitrogen into the plasma facing materials will result in a dynamic modification of the surface structure and composition during the operation of a fusion reactor. Mixing of materials might severely influence physical properties of the plasma facing components, such as the sputtering yield, the melting point or the electrical and thermal conductivity. It is therefore of fundamental interest to fusion research to study the formation and the properties of such multi-component surfaces.

In a recent study of Schmid et al. it was found that for high fluences of energetic nitrogen ions impinging onto tungsten surfaces, a tungsten-nitride surface layer is developed within the ion penetration depth [3]. Dynamic equilibrium conditions for the surface composition are established, after the removal of a surface layer with a thickness of the order of the ion range. The authors also report that due to the accumulation of nitrogen in the surface, the partial tungsten sputtering yield is reduced as compared to sputtering of pure tungsten surfaces. From this point of view the formation of tungsten nitride mixed material layers in a fusion reactor could in fact be beneficial by decreasing the tungsten impurity release into the plasma.

It was also found [4] that the resistivity of tungsten nitride films varies with the deposition conditions. It increases with the nitrogen content in the surface and can be as high as 3000–5000 $\mu\Omega$ cm for as-deposited, nitrogen rich films. While for conducting targets the sputtering yield generally only depends on the kinetic energy

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of the projectile [5,6], a pronounced enhancement of the erosion rate with the charge state of the impinging ion was found for some insulating and semi-conducting targets, like alkali halides [7,8], oxides [5,9] and also hydrocarbon surfaces [10]. This phenomenon is generally referred to as 'potential sputtering' ([11] and references therein). It is mainly observed for target materials with a strong electron–phonon coupling, where the electronic excitation, which is introduced into the surface by the deposition of potential energy (i.e. the sum of the ionization energies) of the highly charged ion during surface impact, can efficiently be converted into motion of target atoms.

In a fusion reactor, elevated sputtering yields of nitrogen containing wall material due to potential sputtering by highly charged impurity ion impact, would give rise to undesirably enhanced wall erosion rates and core plasma contamination by high Z impurities. This study therefore aims at testing whether nitrogen saturated tungsten surfaces are prone to potential sputtering under subsequent impact of highly charged noble gas species such as argon or neon ($Ar^{q+}$ and $Ne^{q+}$, respectively), which are both considered to be used as seeding impurities in future fusion reactors like ITER (International Thermonuclear Experimental Reactor). We conducted our investigations under controlled laboratory conditions, which enabled us to investigate the sputtering yield for mono-energetic particle impact of a single ion species with a well-defined charge state.

## 2. Experimental setup

Total sputtering yields of a tungsten nitride surface under the impact of seeding ions were determined using a highly sensitive quartz crystal microbalance (QCM) setup [12–15] connected to the electron cyclotron resonance (ECR) ion source facility at the Technical University of Vienna (TU Wien) [16]. A sketch of the experimental setup is given in Fig. 1. The mass sensitive part of the setup is a plano-convex, stress compensated cut quartz crystal, which is driven at its fundamental oscillation in thickness shear mode. It is operated at its resonance frequency of ~6 MHz by applying an AC voltage to the gold electrodes on both sides. The QCM is mounted on a linear translation stage in an ultra high vacuum (UHV) chamber at a base pressure in the low $10^{-10}$ mbar regime. The beam facing side of the quartz crystal is coated with a 500 nm thick, polycrystalline tungsten film. The tungsten surface is deposited onto one of the gold electrodes of the crystal by vapor deposition in a separate preparation chamber and transferred to the experimental chamber ex vacuo. The total mass change of the target film is reflected in a change of the resonance frequency according to the relation $\Delta f/f = -\Delta m/m$ [17]. Accordingly this method does not allow determining the actually sputtered species, nor is it possible to disentangle any simultaneous mass loss (e.g. erosion) and mass increase (e.g. projectile implantation).

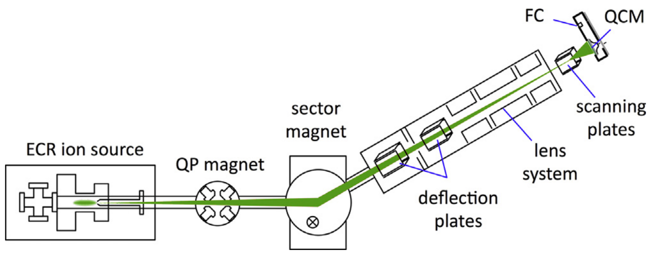

**Fig. 1.** Sketch of the experimental setup including the ECR ion source for the production of highly charged ions, a pair of quadrupole magnets for focusing the ion beam onto the sector magnet, where the ions are separated according to their $m/q$ ratio and finally the beam line, which consist of an ion optics (with two pair of deflection plates and an einzel-lens), a pair of scanning plates and the QCM sample holder.

A sophisticated electronics enables us to detect mass changes of as small as $10^{-5}$ μg/s [13]. In addition all experiments are conducted at an elevated temperature of 465 K, where temperature fluctuations of the quartz due to the ion impact have a minimal influence on the resonance frequency. A more detailed description of the QCM setup can be found in Refs. [12,13].

The projectile ions $N^+$, $Ar^{q+}$ and $Ne^{q+}$ ($q = 1$–9) are produced in the 14.5 GHz ion source SOPHIE of the ion beam facility at TU Vienna [16]. After extraction they are focused onto the entrance of a sector magnet by a pair of quadrupoles and then mass over charge state selected in the sector field. The resulting ion beam is deflected and focused onto the sample position by means of an electrostatic lens system and deflection plates (cf. Fig. 1). A 3 and a 5 mm aperture are used for beam collimation and allow producing a beam spot size of approximately 2 mm after focusing onto the sample position. Right in front of the surface, the ion beam is scanned over the whole active area of the crystal (i.e. an area of ≥7 mm in diameter) by applying two AC signals of different frequency to a set of scanning plates. Thereby a homogenous beam coverage and an accurate determination of the ion current density contributing to the detected mass change can be guaranteed and mechanically imposed stress can be kept at a minimum [12,13]. The ion current density impinging onto the sample is determined by means of a faraday cup, which is located at a given distance above the quartz crystal on the sample holder. Ion current measurements are conducted before and after each sputtering yield measurement.

Nitrogen saturated tungsten surfaces are prepared by implanting energetic nitrogen ions into a freshly deposited tungsten surface. The nitrogen saturated tungsten layers are not annealed after implantation and are further investigated without any additional treatment or analysis. The nitrogen impact energy and saturation fluence were chosen such, as to ensure that the $Ar^{q+}$ and $Ne^{q+}$ projectiles used in subsequent investigations, predominantly interact with the nitrogen saturated surface layer along their path through the surface. For this purpose ion ranges of all three projectiles were estimated with the Monte Carlo code TRIDYN [18]. TRIDYN is a code, which calculates kinetically induced sputtering yields and ion ranges in a binary collision approximation. It simulates the trajectories of the projectile and the recoiling target atoms as a series of two body collisions. TRIDYN is in principle also capable of considering compositional changes of the surface, resulting from the applied projectile fluence. For estimating the projectile ranges however, static simulations were performed in this case, i.e. any compositional changes of the surface were suppressed. The implantation profile of N was estimated for a pure W surface; ranges of Ne and Ar were determined on the resulting W-nitride surface. For this purpose an atomic fraction of W/N = 1 was assumed in the calculations, which is in accordance to the surface composition found in the studies of Schmid et al. [3]. The surface binding energy of both, the simulated W and the WN surface was set to the value generally used for pure W, i.e. 8.68 eV [19].

The energy of the $N^+$ ions, which were used for saturating the W surface, was adapted with respect to obtaining a similar N implantation profile as compared to the ion range profiles of the noble gas ions (Ar and Ne) at the highest energy subsequently investigated in the sputtering experiments. Consequently, the impact energy for $N^+$ ion bombardment was set to 4000 eV and the sputtering investigations for Ne and Ar projectile ions were conducted only below an impact energy of 4900 eV. In Fig. 2 the implantation profile as obtained from TRIDYN of 4000 eV $N^+$ ions in W (in green) is compared to the profiles of $Ar^+$ (in red) and $Ne^+$ ions (in blue) in a WN surface at the highest investigated energy of 4900 eV.

For obtaining a N saturated W-nitride surface, the $N^+$ irradiation was continued up to a cumulated ion fluence of $\sim 3.5 \times 10^{17}$ N/cm$^2$. The mass change of the W surface under ion impact was monitored throughout the entire saturation process. With increasing

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