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Interaction of nitrogen ions with beryllium surfaces

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ABSTRACT

The interaction of energetic nitrogen projectiles with a beryllium surface is studied using a highly sensitive quartz crystal microbalance technique. The overall mass change rate of the beryllium sample under N_2^+ ion impact at an ion energy of 5000 eV (i.e. 2500 eV per N) is investigated in situ and in real-time. A strong dependency of the observed mass change rate on the nitrogen fluence (at constant flux) is found and can be attributed to the formation of a nitrogen-containing mixed material layer within the ion penetration depth. The presented data elucidate the dynamics of the interaction process and the surface saturation with increasing nitrogen fluence in a unique way. Basically, distinct interaction regimes can be discriminated, which can be linked to the evolution of the surface composition upon nitrogen impact.

Steady state surface conditions are obtained at a total cumulative nitrogen fluence of $\sim 80 \times 10^{16}$ N atoms per cm^2. In dynamic equilibrium, the interaction is marked by continuous surface erosion. In this case, the observed total sputtering yield becomes independent from the applied nitrogen fluence and is of the order of 0.4 beryllium atoms per impinging nitrogen atom.

1. Introduction

The international experimental thermonuclear fusion reactor ITER is currently being built in Cadarache in the south of France. Its aim is to demonstrate the usability of fusion for power generation. ITER will be the first fusion reactor to ever deliver a net energy output, with a scientific goal of producing ten times the power that is needed as auxiliary heating power in order to sustain the fusion reaction (i.e. a fusion power gain of $Q \sim 10$).

As a result of the extreme conditions, which are necessary for the operation of a future fusion power plant, the interaction of the very hot fusion plasma with the walls of the vacuum vessel will be a major challenge. A profound knowledge about the key plasma-wall interaction issues such as surface erosion, implantation of impurities and retention of the fuelled hydrogen isotopes is therefore strongly desired.

In ITER a material mix of beryllium on the first wall and tungsten in the divertor at the bottom of the machine is envisaged. ASDEX Upgrade was the first fusion machine to operate with a full tungsten wall [1] and also JET has recently commenced operation with an ITER-like wall composed of both tungsten and beryllium [2,3]. With the transition to all-metal machines, the intrinsic impurity levels (carbon) in the plasma edge decrease considerably and their beneficial influence on heat load mitigation by radiative cooling is reduced [4]. In order to maintain the desired radiated power fraction in high power discharges, a controlled injection of impurity species to replace intrinsically available coolants will therefore be mandatory [5]. For this purpose, nitrogen has proven to be a valuable substitute for e.g. carbon in the divertor region, due to its favorable radiative characteristics at low electron temperatures [1,6]. Nitrogen seeding with feedback control has already matured into a standard operational scenario in ASDEX Upgrade [4]. With nitrogen as a seeding gas, the divertor target power flux could be mitigated by high radiation to a technically acceptable level, and the plasma performance could even be increased compared to discharges without impurity seeding [1,7].

In a series of recent laboratory experiments it was observed that under nitrogen impact, a stable nitride phase is formed within the penetration range in both ITER plasma wall materials, i.e. a WN_x [8] compound and Be_3N_2, respectively [9]. Compositional changes of the plasma facing components might have a severe influence on material properties such as the sputtering yield, the melting point, the fuel retention behavior or the electrical conductivity of the

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surface. The evolution of plasma facing materials like tungsten and beryllium in the environment of a burning fusion plasma, especially the mixing with impurity and fuelling species, is therefore of considerable interest.

In this work the saturation dynamics of a beryllium surface under nitrogen impact was studied in detail. The investigations were performed under controlled laboratory conditions using a mono-energetic nitrogen ion beam. Mass change rates were determined in situ and in real-time.

A unique insight into the dynamics and built-up of mixed nitrogen-containing beryllium layers could be gained, which can be a valuable contribution for understanding the basic mechanisms of the involved processes. In addition steady state surface conditions were also studied, where the simultaneous formation and erosion of the resulting modified surface layer is in dynamic equilibrium.

2. Experimental approach

Total mass change rates of a beryllium surface under nitrogen impact were studied at IPP Garching using a highly sensitive quartz crystal microbalance technique developed at the University of Technology in Vienna [10–12]. The centerpiece of the setup is a stress compensated (SC) cut quartz crystal, which is driven at its thickness shear mode in series resonance at a resonance frequency of about 6 MHz.

One side of the quartz crystal is coated with a 500 nm thick beryllium layer. The beryllium layers are fabricated by thermionic vacuum arc deposition at the National Institute of Laser, Plasma and Radiation Physics in Bucharest [13]. Dense beryllium layers with a density close to that of bulk beryllium (i.e. 1.24×10^{26} Be/m^3) are produced by accelerating the beryllium ions onto the gold substrate by means of a negative bias of – 75 V. The substrate temperature during deposition is kept constant at 473 K. The beryllium films are transferred in air before being installed in a vacuum chamber for exposure to a nitrogen ion beam. The beryllium samples used in the presented studies therefore exhibit a native oxide layer on the surface.

Total mass changes of the target film under nitrogen impact are determined from a change in the resonance frequency of the quartz crystal according to the relation $\Delta f/f = -\Delta m/m$ [14]. In our experiments any mass change rate is therefore derived from the slope of a linear fit to the frequency-change curve with fluence. In cases where the mass change rate changes with the cumulative ion fluence, a step-wise linear approximation is used to evaluate the mass change rate of the target film. For this purpose the resonance frequency of the quartz is usually recorded once every 500 ms.

The quartz crystal microbalance (QCM) technique exploited in our investigations is by design only capable of detecting total mass changes, but does not deliver any information on the actually sputtered species. In addition, any simultaneous mass increase $+\Delta m$ (due to e.g. projectile implantation) and mass loss $-\Delta m$ (by e.g. material erosion) cannot be distinguished. Thus only the balance of all processes, which result in a mass change of the target film, is observable.

The quartz crystal is mounted on a sample holder, which is depicted in Fig. 1. The beryllium-coated quartz (①) is clamped onto the sample holder by a tungsten spring from the back (②) and with a gold ring from the front (③), in order to avoid mechanically induced stress. The sample holder is equipped with a faraday cup (④) for frequent ion current density determination in between surface exposures to the impinging nitrogen beam. A linear translation stage allows for changing between quartz and faraday cup position.

Experiments are performed in an ultra high vacuum chamber at a base pressure of approximately 1×10^{-9} mbar. The chamber is pumped by a combination of a turbo-molecular pump, a titanium sublimation pump and a liquid nitrogen baffle, which is cooled with liquid nitrogen during the experiments.

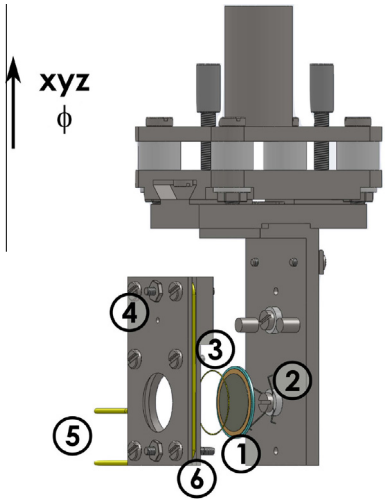

Fig. 1. Sketch of the sample holder, including: the quartz crystal ①, the crystal clamping from the back with a W spring ② and from the front with a Au ring ③, the Faraday cup ④ for ion current density determination, a coaxial heater ⑤ and thermocouple ⑥ to keep and monitor the quartz operating temperature.

In order to minimize influences of temperature changes during ion bombardment on the resonance frequency of the quartz crystal, measurements were always conducted at an elevated surface temperature of 465 K, which is expected to be a typical ITER first wall temperature [15]. At this temperature the resonance frequency of the quartz is least sensitive to temperature fluctuations. The crystal is heated with a constant heating power by means of a coaxial heater (⑤ in Fig. 1), clamped onto the sample holder. The temperature is monitored by a k-type thermocouple attached to the back of the sample holder in near vicinity to the quartz (at ⑥ in Fig. 1). Sophisticated electronics permits the detection of mass changes of as small as 0.2 amu/ion μA/cm^2 [11,12], corresponding to approximately 5×10^{-5} beryllium monolayers per second or 3×10^{-6} amu per ion for typical ion current densities characteristic to the experimental conditions in the presented investigations.

A schematics of the experimental setup is shown in Fig. 2. Molecular N_2^+ ions are produced in a commercially available electron impact ion source (SPECS IQE 12/38). The source is operated at a pressure of $\sim 1–5 \times 10^{-5}$ mbar, which results in a pressure increase to about 10^{-7} mbar in the experimental chamber during source operation.

A Wien velocity filter is used to select the molecular ions from the extracted ion beam. The ion source and the direction of the ion beam when it exits the Wien filter, are slightly off axis in order to keep neutral particles from passing the setup. The ions are accelerated from the ion source with a voltage of 5000 V, resulting in a surface impact energy of 2500 eV per constituent nitrogen atom. By means of two lenses and a pair of deflection plates, the extracted ion beam is then guided and focused onto the sample position in the experimental chamber.

The oscillation amplitude of the quartz decreases from the center outwards, which introduces a radial dependency of the sensitivity of the quartz to mass changes (energy trapping [14]). To eliminate this effect in the determination of mass change rates from the observed frequency evolution of the quartz, the whole mass sensitive inner part of the crystal (the so-called active area) has to be covered with a homogenous ion current density. Therefore the impinging ion beam is scanned rapidly across the entire active area (i.e. an area exceeding 7×7 mm^2) by applying two