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Том 3

STUDYING PLASMA-WALL-INTERACTION PROCESSES IN THE LABORATORY USING A SENSITIVE QUARTZ CRYSTAL MICROBALANCE

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In nuclear fusion research, ion-solid interaction processes between plasma ions and relevant first wall materials like W are under continuous investigation. During operation of a magnetically confined nuclear fusion device, erosion of the reactor's first wall is occurring due to the continuous bombardment by energetic ions, called sputtering. This process is disadvantageous since sputtered wall atoms penetrate the fusion plasma, increase radiative losses and thus reduce the plasma temperature. Furthermore, retention of radioactive tritium due to ion implantation in the first wall has critical safety issues [1]. Since W has a comparably low sputtering yield and low hydrogen solubility, it is a promising candidate for application in fusion devices [2]. However, precise determination of material properties by laboratory experiments is crucial for the development of future nuclear fusion reactors.

In plasma-wall interaction research facilities, linear plasma devices are often used to mimic ion bombardment on materials inside an operating nuclear fusion device [3]. These experiments allow reaching similar ion fluxes and fluences like expected for the fusion reactor ITER [4]. However, the intense conditions can also form limitations for very filigree measurements, which may demand better control of experimental parameters and dynamic processes.

At TU Wien, we therefore established an experimental setup for investigation of first wall relevant material properties with special focus on parameter control. A simplified scheme of the setup is provided in figure 1. The key feature is the implementation of a highly sensitive Quartz Crystal Microbalance (QCM) technique in combination with an electron ionisation ion source in an UHV chamber, which allows measuring very small mass changes of a sample under ion bombardment *in-situ.* For this approach, the samples need to consist of quartz crystals coated with a thin layer (several 100nm) of the desired material of interest. During the experiments, a dedicated electronic circuit allows to record the resonance frequency of this quartz crystal during ion bombardment, which is directly connected to the mass change [5, 6]. For the special case of W, detection of erosion rates as small as 10^{-4} monolayers per second are possible, which is especially useful for precise investigation of materials with relatively small sputter yields [7]. A Faraday cup allows to measure ion beam fluxes, which are necessary for sputter yield determination. Our ion source (SPECS IQE 12/38) enables a precise, liberate choice of ion energy, and is furthermore equipped with a Wien-filter module that allows both to choose a certain ion species while excluding transmission of energetic neutral particles. In addition, the flux of ions can be varied in order to mitigate dynamic modification of irradiated samples if desired. A recent experimental upgrade furthermore allows us to control the Wien-filter remotely via PC. In combination with the opportunity to supply selected working gas mixtures to the ion source, more complex irradiation procedures are possible, i.e., using a fast variation of ion species. By exploiting two sequential differential pumping stages, which are realised at the Wien-filter and at the deflection plate module, influences of the working gas on the background pressure are kept to a minimum. Usual pressures during operation are in the E-9 mbar range for Ar^{1+} irradiation. Further systematic manipulation of the background pressure can be performed using an additional fine tunable leak valve connected to a laboratory bottle with desired gas mixture, which allows adsorbate dependent sputter yield measurements.

The implementation of the so-called 3rd mode QCM technique [7] stipulates another key upgrade of the experimental setup. For this method, both the casual ground resonance and the third overtone frequency of the quartz crystal are recorded, which allows to determine a beat frequency that is only dependent on temperature variations and not on mass changes [7]. Using this quantity in our measurements, a convenient method for measuring actual quartz crystal temperatures without need of a thermocouple is available. Moreover, this technique also allows to compensate any temperature related effects on the recorded signal. An application of this method is important for determination of small sputtering yields like i.e., for D ions on W targets, where laboratory temperature variations can significantly contribute to the recorded frequency signal. Furthermore, this compensation technique allows to operate the QCM also at elevated temperatures using an ohmic heating system and to investigate outgassing processes of retained ions. For the latter purpose, a quadrupole mass spectrometer (Pfeiffer Vacuum PrismaPlus) enables to perform Thermal Desorption Spectroscopy (TDS) measurements, while the total mass loss during the temperature increase can be recorded using the 3rd mode QCM technique [7].

Since an application of the QCM requires special samples consisting of quartz crystals with a thin coating of the desired material, experiments in the past were limited to rather idealistic surfaces with smooth textures only. In order to liberate this constraint, a so-called

Catcher - QCM (C-QCM) apparatus was implemented to the experimental setup. Based on the work of Berger et al. [8] and by usage of a manipulator with four degrees of freedom, our C-QCM can be freely positioned with respect to the irradiated sample. The C-QCM is used to investigate the angular distribution of sputtered atoms from the target, which leads to a net mass increase on the catcher crystal. Therefore, any kind of target samples, i.e., plain bulk sheets of W metal, advanced coatings with special surface structure but also full pieces of plasma facing components from prior studies in real nuclear fusion devices can be investigated in our setup.

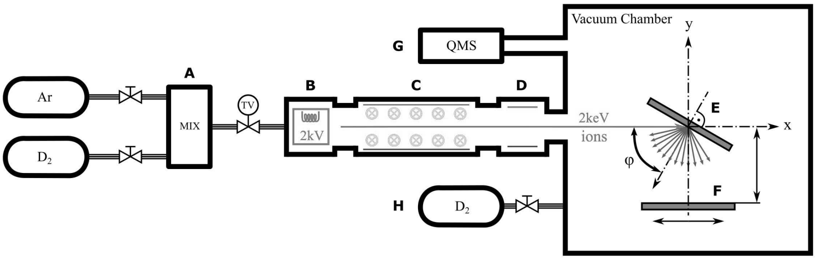

Figure 1: Simplified scheme of the QCM Setup at TU Wien. A - Reservoir for working gas mixture; B - Electron impact ion source (SPECS IQE 12/38) supported by a thermovalve (TV); C - Wien-filter with remote control for ion selection; D - Deflection plates for beam scanning; E - Target QCM irradiated under ion incidence angle φ; F - Catcher QCM; G - Quadrupole mass spectrometer; H - Fine-tunable leak valve.

An exemplary demonstration of the strengths and capabilities of our QCM setup is presented in figure 2. Here, two individual C-QCM measurements were performed using both a comparably smooth W film with 29 nm RMS roughness for comparison and a bulk sample with complex W fuzz morphology under Ar^{1+} irradiation and an angle of incidence of 60° (respective target surface normal). W fuzz, which is a fibrous network on the nm-scale, develops under special irradiation conditions within a certain surface temperature regime and He plasma exposure [9]. Recent studies revealed that virgin W fuzz structures show severe reduction in effective sputtering yields by a factor of 17 in comparison to flat W surfaces due to enhanced local redeposition in this fibrous network [10]. With continuing erosion of the W-fuzz sample (i.e., by applying a certain Ar^{1+} fluence), the measured yield increases again but does not reach that of a smooth sample [10]. SEM measurements of the fuzz samples after irradiation show the formation of a scaly structure facing in the direction of the incident ion beam [10]. In figure. 2, results for such a W fuzz sample are shown after application of a total Ar^{1+} fluence of 2.3×10^{20} Ar/m² (corresponding to approx. 7 nm erosion) and compared to the measured trend for a smoother W film. Sputtering of the pre-irradiated fuzz is about 50 to 60% lower over the whole investigated angular range than for the smooth W target. This indicates that a considerable reduction in sputtering yield remains after alteration of the initial fuzz structure. These results are in good agreement with previous measurements [10].

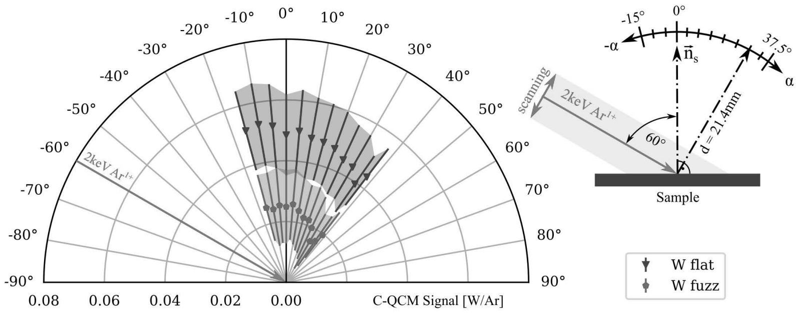

Figure 1: Polar plot of the Catcher-QCM signal, measured along a circular path around the target. Experiments were performed with 2keV Ar^{1+} ions under 60° ion incidence for both a smooth W film (blue) and for a pre-irradiated W fuzz sample (green). The positive signal values correspond to a net mass increase on the Catcher-QCM due to deposition of sputtered atoms from the target. The shaded areas illustrate experimental standard deviations. The sketch in the top right corner highlights the geometrical conditions of the experiment. The lower signal for the pre-irradiated W fuzz sample indicates a reduction of the sputter yield by a factor of 2. Pre-irradiation of the W-fuzz target with Ar^{1+} ions can modify the original fuzz structure into a scaly structure (see [10]).

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