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Sputtering of iron, chromium and tungsten by energetic deuterium ion bombardment

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ABSTRACT

Sputtering of the pure materials iron (Fe), chromium (Cr) and tungsten (W) due to energetic deuterium (D) ion bombardment was investigated. These materials are important constituents of reduced-activation ferritic-martensitic steels. Sputtering yields were measured as a function of the D ion energy from 60 to 2000 eV/D. The obtained data can be well reproduced by a semi-empirical formula suggested by Bohdansky, and the corresponding fitting parameters are provided. It is confirmed that analytical formulae suggested by Eckstein and Yamamura agree satisfactorily with these experimental data. By comparison with results from the binary-collision-approximation-based calculation codes SDTrimSP and SRIM it is found that SRIM has some limitations in simulating sputter yield close to the threshold whereas SDTrimSP results show good agreement with measured data in the investigated energy range.

1. Introduction

Sputtering of plasma-facing materials due to interaction with energetic ions (particularly hydrogen isotopes) is an essential issue in magnetically confined fusion devices because it is directly related to impurity generation as well as to the lifetime of plasma-facing components [1]. Sputtering behavior of candidate materials, such as beryllium and carbon, due to energetic hydrogen isotope ion bombardment was extensively studied in the last several decades [2]. Reduced-activation ferritic-martensitic (RAFM) steels, such as EUROFER [3], RUSFER [4], the Japanese alternative F82H [5] or the Chinese CLAM [6] which are being developed as structural materials for fusion applications, are recently also considered as a possible option for certain areas of plasma-facing surfaces in a future power plant because of technological and economic advantages [7]. This has triggered the evaluation of EUROFER steel erosion by energetic deuterium (D) bombardment [8]. Sputtering of RAFM steel is more complex than for pure elements because steel is a compound material. For example, one can theoretically expect that lighter alloyed elements will be preferentially sputtered, leading to a continuous change of the surface stoichiometry during ion irradiation until a steady state is reached. For a better understanding of the sputtering processes on RAFM steels it is in a first step necessary to know the sputtering of each alloyed element as a reference. However, sputtering data for these elements are still quite limited. A few data for iron and tungsten exist, but for chromium no experimental data are available.

In this study we, therefore, measure the sputtering yields of some of the key elements for RAFM steels, i.e. iron (Fe: the base material), chromium (Cr: the second major alloyed element (~ 10 at.%)) and tungsten (W: the highest-Z admixed element in RAFM steels), under well-defined conditions in order to obtain comprehensive data sets for these constituents. Particular emphasis was put on measuring data close to the threshold for physical sputtering. Thin sputter-deposited films were used in this study because they offer the principal advantage to measure sputter yields with higher sensitivity. For thin films the change in layer thickness after sputtering can be measured with ion beam analysis methods. This procedure allows measuring yields with higher sensitivity than, e.g., weight loss measurements. In this work, results from weight loss measurements are compared with those from ion beam analysis. The obtained data are evaluated with fitting formulae for a parametrization and analytic description of the measured sputtering yields. Furthermore, the experimental data are compared with existing sputtering simulation codes for benchmarking. This step is essential because the erosion rate of steel walls in future fusion devices will be eventually assessed numerically by using such simulation codes.

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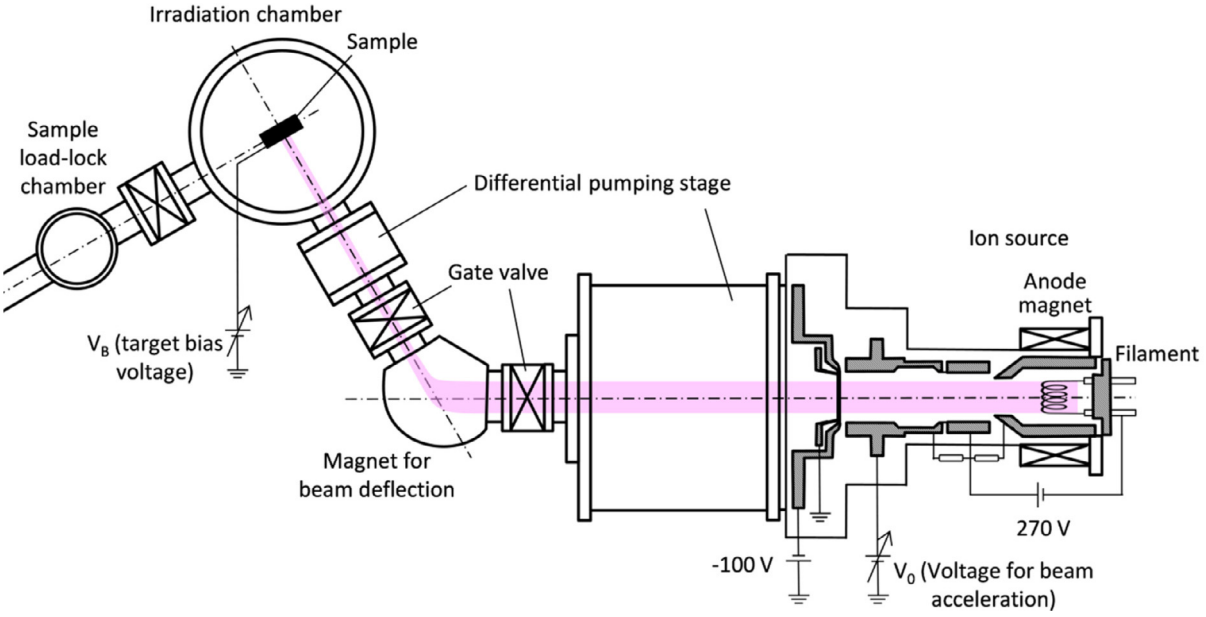

Fig. 1. Schematic view of the high-current ion source set-up [9].

2. Experimental procedure

2.1. Sample preparation

Thin layers of Fe, Cr and W were prepared by magnetron-sputter deposition using a UNIVEX 450B device (Leybold Vacuum GmbH). Single-crystalline silicon (Si) wafers were used as substrates. The sample dimensions were 12 mm × 15 mm. In order to ensure the layer adhesion the Si substrate surface was cleaned by argon (Ar) RF plasma etching for 1 min. prior to the layer deposition. Ar was also used as working gas during deposition at a pressure of 0.3 Pa. The background pressure inside the deposition chamber is $\sim 2–3\times10^{-5}$ Pa. A high power DC-discharge was applied for the magnetron-sputtering of the target (600 W for Fe and Cr, and 300 W for W deposition). The deposition rate was roughly 20 nm/min and the thickness of the deposited layers was 350–400 nm. No bias voltage was applied to the substrate holder. Under these deposition conditions the Ar content in the deposited layers is below the detection limit of the applied ion-beam analysis (see below). In each deposition run a graphite substrate was coated together with the Si substrates. This allows the measurement of low-Z impurities, such as oxygen (O), in the layers by Rutherford backscattering. The determined O concentrations in the layers were about 2–3 at.% for Fe and W and about 5–6 at.% for Cr.

2.2. Deuterium ion irradiation

Prepared specimens were then irradiated by D ions in the high-current ion source set-up (HSQ) at IPP Garching [9]. The HSQ set-up consists of a duo-PIGatron type ion source [10], two differential pumping stages, a sector magnet for beam deflection and a target irradiation chamber connected to a load-lock chamber, as schematically shown in Fig. 1. The sector magnet enables to provide a mass-separated D ion beam at defined ion energy, which is well suited for well-defined sputtering yield measurements. The D energy can be controlled by the ion acceleration voltage and the sample biasing. The dominant ion component generated in the ion source is D_3^+. This ion was chosen as the bombarding species to achieve higher particle fluxes. These molecular D_3^+ ions are considered to be identical to 3 individual D ions impinging with the same velocity as the molecular ion. Correspondingly, the energy per deuteron is 1/3 of the experimentally applied ion energy and the flux is three times the measured ion flux. In this study, the sputtering yield was measured in the D energy range from 60 to 2000 eV/D. The ion beam incident angle was normal to the sample surface.

The ion bombardment induces some change of the surface morphology resulting in the appearance of a visible “footprint” of the D ion beam. The ion beam spot area was determined by measuring the footprint size. It varies from 0.3 to 0.85 cm^2 depending on the D energy. The experimental ion fluxes and fluences were calculated from the measured ion currents and beam spot areas. This includes the implicit assumption that the beam intensity is relatively homogeneous across the beam spot. In fact the variation of irradiation beam intensity was checked by measuring the lateral erosion profile after the D irradiation by scanning the ion-beam analysis beam spot over the sample. The ion-beam analysis beam spot size is about 1 mm^2 and, therefore, significantly smaller than the D irradiation beam spot. The such-determined variation of the current density over the beam spot is of the order of 10 to 20 %. The determined area size is expected to include 10 – 20 % of measurement error. This uncertainty of the beam flux and profile affects the determination of the local beam flux and fluences and the evaluation of the RBS data (see below) but not the evaluation of the weight loss measurements. The ion beam current at the target is typically $\sim 10^{-5}$ A, corresponding to a deuteron flux of $\sim 10^{19}$ $Dm^{-2}s^{-1}$. The irradiation fluences in this work were varied in the range of $1–3\times10^{23}$ Dm^{-2} corresponding to exposure durations between 3 and 9 h. Since the background pressure in the target irradiation chamber is sufficiently low ($\sim 10^{-6}$ Pa), surface oxidation during irradiation is not expected. The sample was not actively cooled during irradiation, resulting in slight temperature rise to 310 up to 360 K depending on the ion impinging energy.

2.3. Post-irradiation analyses

The sputtering yield was evaluated by weight-loss (WL) technique and Rutherford Backscattering Spectrometry (RBS). For WL, the sample weight was measured ex-situ before and after D irradiation by a microbalance system (Sartorius MC21S) having a weight resolution of 1 μg and the measurement uncertainty of ± 3 μg. The sputtering yield was then calculated from the weight loss and the