

Influence of Thin Surface Oxide Films on Deuterium Release from Tungsten

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Due to the affinity of tungsten (W) for oxygen, a natural 2-3 nm thick surface oxide film is almost always present on W samples used in ex-situ laboratory experiments, but will most likely not be present in a fusion reactor, where it would quickly be eroded by the plasma. Therefore, any effects that a natural surface oxide film might have on the interaction of deuterium (D) with W, would represent an unaccounted discrepancy between laboratory experiments and real conditions in a fusion reactor.

We investigated the possible effects of thin (5-100 nm) surface oxide films on D release from W by deliberately oxidizing W samples to thicknesses greater than the natural oxide. We monitored the D content and depth profile within the first 7 μm of these samples with nuclear reaction analysis (NRA), the thermal release of D through these films with thermal desorption spectroscopy (TDS) and the surface modifications of the oxide film with scanning electron microscopy (SEM). We applied a novel approach to address the influence of W oxide on D transport by preparing W samples with a dedicated amount of D directly underneath the surface oxide film. In a first step, we irradiated W samples by 20.3 MeV W ions to create a 2.3 μm thick, defect rich layer of “self-damaged” W. This layer has a high D retention and serves as a D “reservoir” for the release experiments. Next, the traps in this reservoir were decorated by exposure to a “gentle” D plasma with an ion energy of < 5 eV per D at a temperature of 370 K and a fluence of 1.5×10^{24} D/m². After the plasma exposure, the total D retention in the self-damaged, 2.3 μm thick region was determined with NRA to be 2×10^{21} D/m² corresponding to 1.7 at. %. In a last step, the sample surfaces were oxidized by an electro-chemical method described by McCargo et al. [1]. This method is applicable at room temperature and thus avoids thermal release of retained D during oxidation. Furthermore, it produces very smooth and homogenous oxide films of reproducible and precisely adjustable thicknesses.

To analyze the outgassing of D and D-containing molecules from W through these films, we applied TDS up to 1000 K. The results of the TDS measurements show that the oxide films significantly influence and alter the release behavior of D from W in several ways. Firstly, with increasing oxide thickness the low-temperature release peak of D is shifted towards higher temperature. Secondly, above 500 K, D is not only released as D₂, but also in the form of heavy water (D₂O and HDO). Obviously, D chemically reacts with the oxide film during outgassing. Above 700 K, heavy water becomes the dominant release channel, as long as enough oxide is available. This is, surprisingly, the case even if the remaining oxide no longer fully covers the surface and only small islands of surface oxide remain. This indicates high surface mobility of the D atoms on W. Thirdly, at a temperature of 500 K that was held constant for 4 hours we observed a constant flux of outgassing D. Thus, the rate-determining step for D release is not thermal de-trapping from the self-damaged W, but the migration through the oxide. Furthermore, NRA measurements show a high D concentration in the oxide film after that hold at 500 K, indicating a high retention in the oxide itself. As a conclusion, it seems likely that even natural oxide films alter the thermal release of D from W by shifting the dominant release channel to heavy water. This has to be considered in the measurements to achieve an accurate quantification of D release – especially for small amounts of D.

[1] M. McCargo, J. A. Davies, & F. Brown, doi: 10.1139/p63-120

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