IT/P3-18 Carbon Erosion Mitigation by Beryllium Layer Formation in ITER

R.P. Doerner 1), K. Schmid 2), M.J. Baldwin 1), R.W. Conn 1), G. Federici 3), A. Loarte 4), J. Roth 2), A. Wiltner 2)

1) University of California at San Diego, La Jolla, CA. 92093-0417 USA

2) Max-Plank-Institut für Plasmaphysik, Garching, Germany

3) ITER-IT Garching Work Site, Germany

4) EFDA-CSU Garching, Germany

e-mail contact of main author: rdoerner@ucsd.edu

Abstract. A beryllium-seeded deuterium plasma is used in PISCES-B to investigate mixed-material erosion and redeposition properties of ITER relevant divertor materials. The experiments are designed to reduce uncertainties in the prediction of tritium retention in redeposited mixed-materials. The beryllium containing plasma simulates the erosion of first wall material into the ITER sol plasma and its subsequent flow toward the carbon and tungsten material located in the divertor region. The beryllium impurity concentration in the ITER divertor plasma is expected to be in the 1 to 10% range. In PISCES, a small (0.15%) beryllium impurity concentration in the incident plasma onto a carbon target is seen to dramatically reduce the carbon chemical and physical erosion rates. The resultant plasma exposed surfaces contain a large fraction of beryllium-rich layers on surfaces outside the plasma column. Tritium codeposited in these beryllium-rich layers is more easily released than tritium codeposited in carbon layers. Beryllium accumulation on tungsten surfaces exposed to plasma containing only a small beryllium impurity concentration (0.1%) is also observed. Finally, the beryllium seeding oven in PISCES-B utilizes a tungsten crucible to hold the hot beryllium. This crucible destructively melted in PISCES-B after only 100 hours of operation and at temperatures that never exceeded 1550°C. The alloy Be₁₂W spears to have formed in the melt zone of the tungsten crucible. The implications of Be-W alloy formation in ITER is described.

1. Introduction

In 2002, a collaboration was established between the USDOE and EFDA focusing on the plasma materials interaction behavior of C/Be/W mixed materials. The experimental plan consisted of a systematic series of experimental exposures of carbon and tungsten samples to deuterium plasma seeded with a controllable amount of beryllium impurity ions. These experiments were conducted in the PISCES-B beryllium facility at UCSD. Sample analysis of the targets and witness plate coupons collected during the exposures was carried out predominantly at the IPP in Garching. Modeling of the experiments and their resultant surfaces were undertaken both within the US and EU plasma facing component communities.

The PISCES-B device [1, 2] has been modified in two ways to accomplish this experimental program. First, in order to seed a controllable amount of beryllium impurities into the plasma column a commercial high-temperature effusion cell from Veeco Applied EPI has been installed on PISCES-B. The orientation of the atom beam emerging from this oven is oriented in such a way that the beam travels perpendicular to the magnetic axis of PISCES-B. Due to the rather slow thermal velocity of the Be atoms, some amount of the beam of atoms is attenuated in the plasma column due to ionization. The beryllium ions are then entrained in the plasma flow toward the target. The concentration of beryllium ions within the plasma column is measured by observing the BeII line emission at 467 nm and can be controlled by varying the temperature of

the oven. Ionization and excitation rate coefficients were obtained from the ADAS data base [3]. The beryllium ionization rates have previously been experimentally verified in PISCES-B [4].

The second modification to PISCES-B involved the installation of a movable witness plate manipulator system to collect redeposited material outside of the plasma column during the sample exposures. The samples used to collect material can be either cooled of heated, so that any dependence of the hydrogen inventory in the redeposited mixed materials on the temperature of the plate during deposition can be determined. The witness plate manipulator system was supplied to PISCES-B by the IPP at Garching.

2. Beryllium Carbon Interactions

In ITER, the beryllium impurity concentration in the divertor plasma is expected to be in the 1-10% range. We find that even a very small beryllium impurity concentration (as low as 0.2%) is sufficient to dramatically reduce the graphite target chemical and physical erosion rates [2]. Figure 1a shows the spectroscopic signature of chemical erosion (CD band emission) from a sample exposed at 200°C while Figure 1b shows the physical sputtering signature (CI line radiation) from a sample exposed at 700°C to deuterium plasma. Data is presented for cases with and without beryllium seeding of the plasma. Weight loss measurements confirm the spectroscopic signature data and indicate a reduction of the total erosion from the graphite samples by more than an order of magnitude when the beryllium concentration in the plasma is as low as 0.2%.

The cause of the reduction becomes apparent during post-exposure surface analysis of the graphite targets [5]. Auger electron spectroscopy of the sample surface exposed at 200°C reveals essentially complete (>90%) coverage of the graphite sample by a thin beryllium layer as long as the Be seeding concentration exceeds 0.1%. Similar effects are observed during exposure of graphite samples at 1000°C, although a slightly higher Be seeding rate (>0.3%) is needed to achieve 90% Be surface coverage. This is due to the larger rate of diffusion of Be into the bulk at the higher temperature, as well as an increase in the Be erosion rate at higher temperature [6].

The formation of beryllium rich surface layers on the graphite targets also affects the redeposited material collected on the witness plate samples. Figure 2 shows the elemental depth profile (obtained using x-ray photo-electron spectroscopy (XPS)) of the re-deposited material collected on a tantalum witness plate during exposure of a graphite sample at 700°C to a 0.1% Be seeding rate plasma [7]. The redeposited material is seen to consist almost entirely of beryllium, with only trace amounts of oxygen and carbon throughout most of the layer. The average deuterium content in this layer is consistent with previous retention measurements in 'clean' redeposited beryllium [8] at low temperature as seen in Figure 3.

While the hydrogen content of beryllium and carbon co-deposited films is found to be similar during room temperature deposition, the hydrogen content in Be films decreases much faster with increasing deposition temperature than that in carbon films. The temperature dependent release of the deuterium from codeposisted material collected during Be seeding experiments is shown in Figure 4. This data is generated from thermal desorption of the witness plate samples from PISCES-B and can be considered typical of the release behavior of the accumulated codeposited material in the ITER divertor during a baking cycle. One can see that during a 400°C bake of the ITER divertor (a temperature that is achievable in the ITER divertor after draining of the collant channels) the D/Be ratio drops to less than 1%. Also shown in the figure

for comparison is the temperature dependent deuterium release from codeposited carbon films [9].

It is interesting to note that the initial temperature of the wall during the deposition of the material affects the release behavior. The deuterium trapped during room temperature collection is released at lower temperature than that collected during higher temperature wall operation. This may be due to the fact that the material structure will vary depending on the deposition temperature, or it may be related to the oxygen content of the films (see Figure 3). Work is continuing in this area to try to isolate the cause of the differences.

Another difference between a beryllium-rich and a carbon-rich co-deposit will be in its location. Beryllium has a high sticking coefficient and will be deposited in locations with line-of-sight views of the erosion location, whereas carbon has been found to migrate into pump ducts, behind tiles and to other non-line-of-sight locations [10].

3. Beryllium Tungsten Interactions

Similar beryllium plasma seeding experiments have begun using tungsten target samples instead of the carbon targets described above. Figure 5 shows the resultant elemental composition of one such tungsten target exposed to deuterium plasma containing 0.1% beryllium impurities. The plasma exposure conditions were; ion flux = $1 \times 10^{22} \text{ m}^{-2} \text{s}^{-1}$, ion energy 75 eV, electron temperature 6 eV, target temperature 300°C and exposure duration 5000 sec. As can be seen in the figure, a beryllium rich layer also forms on the tungsten target plate.

It is important to note that the ratio of the beryllium to tungsten at the surface is 12:1. The reason for the importance of this ratio is evident from the Be-W phase diagram [11] reproduced in Figure 6. The phase diagram shows the existence of two beryllium rich alloys, $Be_{22}W$ and $Be_{12}W$. One can speculate this alloy forms in the surface region of the tungsten targets exposed to the beryllium containing plasma. The reason that the formation of such an alloy is important is because this alloy has a melting temperature of approximately 1500°C, much lower than that of the tungsten substrate material.

At present, PISCES-B cannot expose targets to plasma at temperatures exceeding approximately 1100°C, so quantitatively examining the behavior of these low melting temperature Be-W alloys is difficult. Some qualitative insight into the importance of Be-W alloys can be gained however, as the effusion cell being used as the Be oven in these experiments utilized a tungsten crucible to hold the high temperature beryllium ingot being used to seed the deuterium plasma with beryllium impurities.

The tungsten crucible used in PISCES-B suffered destructive melting after only about 100 hours of beryllium seeding operation. While the temperature of the tungsten crucible varied from day to day, its maximum operating temperature never exceeded 1550°C. The actual failure of the crucible occurred during operation at only 1200°C.

Fragments of the disintegrated crucible have been analyzed using the wavelength dispersive spectroscopy (WDS) system attached to our scanning electron microscope. Two images are shown in Figure 7. Figure 7a shows a fragment of an intact region of the tungsten crucible wall. In this region a thin beryllium rich layer containing approximately 4% tungsten is observed to cover the inner wall of the tungsten crucible. This concentration is consistent with the Be₂₂W alloy. Figure 7b shows a similar image of a section of the tungsten crucible from the failure region. Clearly, a much different, nodular structure appears in this region. The compositional analysis reveals 9%W distributed in a beryllium rich material extending throughout the remnants

of the crucible wall. This composition is similar to the $B_{12}W$ alloy represented in the Be-W phase diagram.

It is difficult to draw any definitive conclusions from the observations of Be-W interactions within the PISCES-B beryllium oven. But based on these observations, it is clear that additional dedicated research is needed in this area. One can say, however, that Be coatings have been observed to form on tungsten samples exposed to beryllium seeded plasma. If such coatings form in ITER, and Be-W alloy formation occurs, the worst case scenario could be that large areas of the tungsten plasma facing components may melt or lose their structural properties.

4. Summary

The current US-EU collaboration has been successful in several areas. First, a dramatic suppression of carbon erosion, both physical sputtering and chemical erosion, has been observed and documented at very low levels of incident beryllium impurity ions. The computer models that are typically being used to predict plasma-facing component lifetimes are currently being modified to attempt to duplicate these experimental findings. Second, the formation of a protective beryllium surface layer over carbon samples impacts the composition of codeposited material eroding from the samples. Material that codeposits away from direct plasma bombardment is composed almost entirely of eroded beryllium. The hydrogen isotope retention behavior of these beryllium-rich codeposits shows that less retention is expected in these codeposits and that it should be easier to remove the retained tritium, as compared to carbon codeposited layers.

Unfortunately, such beryllium coatings have also been observed to form on tungsten plasmafacing components and might be expected to form under ITER conditions. Indications are that these coatings could result in low-melting point Be-W alloy formation. A systematic study of beryllium tungsten alloys is urgently needed.

References

[1] Goebel, D. M., et al., J. Nucl. Mater. **121**(1984)277.

[2] Doerner, R. P., et al., Physica Scripta T111(2004)75.

[3] Summers, H. P., Atomic Data and Analysis Structure – User Manual, Rep. JET-IR(94), JET Joint Undertaking, Abingdon (1994).

- [4] Doerner, R. P., et al., J. Appl. Phys. 93(2003)5816.
- [5] Schmid, K., et al., Nucl. Fusion 44 (2004) 815.
- [6] Doerner, R. P., et al., J. Appl. Phys. 95 (2004) 4471.
- [7] Baldwin, M. J., et al., J. Nucl. Mater. (in press)2004.
- [8] Causey, R. A. and Walsh, D. S., J. Nucl. Mater. 254(1998)84.
- [9] Causey, R. A., et al., J. Nucl. Mater. 176&177(1990)987.
- [10] Federici, G., et al., Nucl. Fusion 41(2001)1967.
- [11] Okamoto, H. and Tanner, L. E., in Phase Diagrams of Binary Tungsten Alloys, Ed. Naidu,

S. V. and Rao, P., Indian Institue of Metals, Calcutta 1991.



Figure 1 – Beryllium impurity seeding of the incident deuterium plasma reduces both a) chemical erosion and b) physical sputtering of graphite target plates.



Figure 2 – Depth profile of the composition of a tantalum witness plate sample used to collect redeposited material during exposure of a 700°C graphite sample to beryllium containing, deuterium plasma.





Figure 3 – Deuterium retention in witness plate samples collected during Be seeded plasma exposures



Figure 4 – Thermal release behavior of codeposited Be films compared to codeposited carbon films.



Figure 5 – Elemental composition of a tungsten target plate exposed to a 0.1% beryllium containing deuterium plasma.



Figure 6 – Be-W phase diagram (reproduced from [11]).



Figure 7a – Cross-section of the tungsten crucible showing a Be-W material coating on the inner wall of the crucible in the region that did not melt.



Figure 7*b* – *An image of a crucible remnant from the crucible melt zone.*