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Tritium Accumulation and Ozone Decontamination of Tungsten and Beryllium

Dominic Batzler^a, Robin Größle^a, Philipp Haag^a, Elizabeth Paine^b, Marco Röllig^a, Marie-Christine Schäfer^a, Marius Schaufelberger^a, and Kerstin Trost^a

^aTritium Laboratory Karlsruhe, Institute for Astroparticle Physics, Karlsruhe Institute of Technology, Eggenstein-Leopoldshafen, Germany; ^bDepartment of Applied Physics, Eindhoven University of Technology, Eindhoven, The Netherlands

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Abstract — Tritium adsorption on surfaces creates a variety of issues ranging from the fields of fusion applications to small- and large-scale laboratory experiments using tritium. The extent to which tritium accumulates on surfaces is generally material dependent and must be determined through experiments. Additionally, this surface contamination necessitates the implementation of appropriate decontamination procedures, preferably in situ. A suitable method could be exposure to ozone during ultraviolet irradiation. However, it is currently not known if both components are necessary for the decontamination. At Tritium Laboratory Karlsruhe, both questions on contamination and decontamination can be addressed using a single experimental setup. With this, it is possible to expose solid samples to gaseous tritium to measure the temporal activity evolution. Furthermore, the system can be filled with dry air and with dry air containing ozone to explore their decontamination effect. Both measurement modes were applied to beryllium and tungsten samples, which were chosen for their relevance in fusion. The beryllium surface was observed to accumulate tritium more than four times faster than tungsten when exposed to gaseous tritium. Concerning the decontamination, without simultaneous ultraviolet irradiation, exposure to ozone did not have any distinct effect on the surface activity compared to simply using dry air.

Keywords — Tritium, Tritium Laboratory Karlsruhe, tungsten, beryllium, beta-induced X-ray spectrometry.

I. INTRODUCTION

Exposure to gaseous tritium often results in activity accumulation on the surface. Such contamination poses multiple challenges for a fusion fuel cycle and for tritium-handling facilities in general. These include the loss of process gas, the accumulation of tritium inventory, the complication of tritium accountancy calculations, and the need for increased safety precautions during maintenance.

In tritium-monitoring systems, this undesired contamination results in a history-dependent, elevated background signal, which is often referred to as memory effect [1]. For neutrino mass experiments, such as the Karlsruhe Tritium Neutrino (KATRIN) experiment [2,3], this creates a systematic effect that needs to be studied and accounted for.

Therefore, suitable decontamination strategies, ideally in situ, are required to mitigate the aforementioned issues. While traditional methods, such as bake-out, are somewhat successful at reducing the surface activity [4], the high temperatures in this case could create additional problems such as increased tritium permeation.

Ultraviolet/ozone exposure presents itself as a promising candidate for in situ decontamination, which has already been proven to be effective on tritiated surfaces, with a decontamination factor of up to three orders of magnitude [5,6]. However, from literature it is

CONTACT Dominic Batzler  dominic.batzler@kit.edu

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not clear which component is responsible for the decontamination efficiency. Some reports suggest that ozone or oxygen radicals alone are sufficient [7,8], while others conclude that only the combination of ozone and simultaneous ultraviolet irradiation are effective [6,9].

To briefly summarize the provided literature, the ozone reacts with surface-bound molecules to form volatile species, which then desorb. Simultaneous ultraviolet irradiation is suspected to dissociate the adsorbed species into fragments, which are more likely to react with ozone, hence increasing the decontamination efficiency. In case only ozone was required, the in situ decontamination would be less complicated to put into practice.

To address these questions, the Tritium Activity Chamber Experiment (TRACE) was employed [4]. It is suited to expose solid samples both to gaseous tritium for contamination and ozone (without ultraviolet irradiation) for decontamination. Both measurement modes were applied to tungsten and beryllium due to their significance for fusion. Beryllium is also of interest for use in the KATRIN experiment. In this work, the results on the surface activity evolution of tungsten and beryllium due to exposure to tritium, as well as the effect of ozone on their surface activity, are reported.

II. EXPERIMENTAL SETUP

The TRACE system consists of a sample cell based on DN 40 CF components with optimized geometry to minimize tritium inventory. A gold-coated beryllium window serves as one of the interfaces and enables in situ measurements of the sample's surface activity by beta-induced X-ray spectrometry (BIXS) [10]. The BIXS system was calibrated to measure the activity by introducing a known quantity of tritium into the recipient. The pressure was chosen sufficiently high such that the X-rays indirectly generated by the decaying gaseous tritium were the dominating contribution to the measured signal. To account for the differing X-ray yields, this procedure was performed for each sample as the last action before completing the contamination campaign. For a more detailed description of the experimental setup, see Ref. [4]. An ultraviolet-based ozone generator, similar to that used in Ref. [11], was connected to the measurement cell to expose the samples to ozone. Throughout the measurement campaigns, the purity of the tritium used for contamination was above 95%.

II.A. Sample Preparation

In this study, three cylinder-shaped (diameter of 33 mm) S-65 beryllium samples by Materion with a

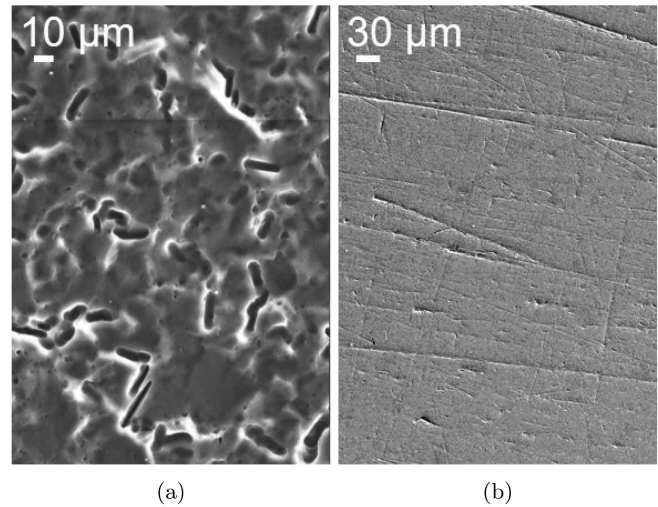


Fig. 1. SEM images of the (a) beryllium and (b) tungsten samples.

minimum beryllium content of 99.2% were used. Because of beryllium's X-ray transmissivity, they possessed a height of 10 mm to prevent measuring tritium below the samples. To avoid generating dust and to minimize the exposure to beryllium, the samples were left unpolished and were only cleaned with ethanol prior to being installed in the measurement cell. A tungsten sample (diameter of 35 mm) was cut from a 1-mm-thick rolled metal sheet with an elemental purity of over 99% according to X-ray fluorescence analyses. Unlike the beryllium samples, the tungsten sample was manually polished and cleaned in an ultrasonic bath. Fig. 1 shows images of the beryllium and tungsten surfaces taken using a scanning electron microscope (SEM), which reveal their different topologies.

Before the start of the measurement campaigns, the samples were baked out at 200°C until a combined leak and outgassing rate of lower than 10^{-9} mbar·l/s was achieved.

III. RESULTS

III.A. Contamination

The samples were exposed to gaseous tritium for 10 min, followed by an evacuation period to measure the remaining tritium on the samples' surfaces in situ using BIXS. By repeating this process, the temporal evolution of the samples' surface activity can be measured. The exposure pressures were selected such that the tritium was in the free molecular flow regime in order to avoid effects from gas self-interaction. For two beryllium and a tungsten sample, a pressure of 10^{-3} mbar was chosen, while a third beryllium sample was exposed to

2×10^{-3} mbar of tritium. Given the system's geometry, these pressures correspond to Knudsen numbers of 30 and 15, respectively.

The time series of the beryllium surface activities are shown in Fig. 2. Both statistical and systematic uncertainties are included in the individual data points; the largest contribution stems from the uncertainty on the sample cell volume for the activity calibration. With each subsequent sample, less activity is accumulated, ranging from a slope of (10.06 ± 0.70) to (2.28 ± 0.22) kBq/(cm²·mbar·s) for the first and third samples, respectively.

With a slope of (0.488 ± 0.026) kBq/(cm²·mbar·s), the tungsten sample retains considerably less tritium on its surface than beryllium, as displayed in Fig. 3. Because of its higher nuclear charge of $Z=74$ compared to beryllium's $Z=4$, the uncertainties are also significantly lower.

III.B. Ozone Decontamination

After concluding the contamination measurements, one of the beryllium and the tungsten samples was used to test the ozone decontamination method. To investigate

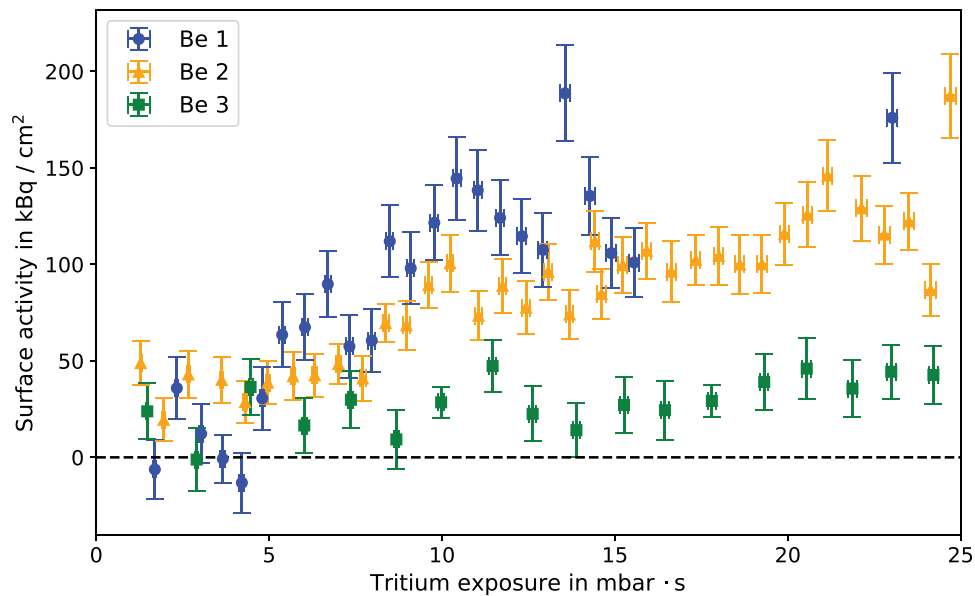


Fig. 2. Increase of surface activity of three beryllium samples due to repeated exposure to gaseous tritium.

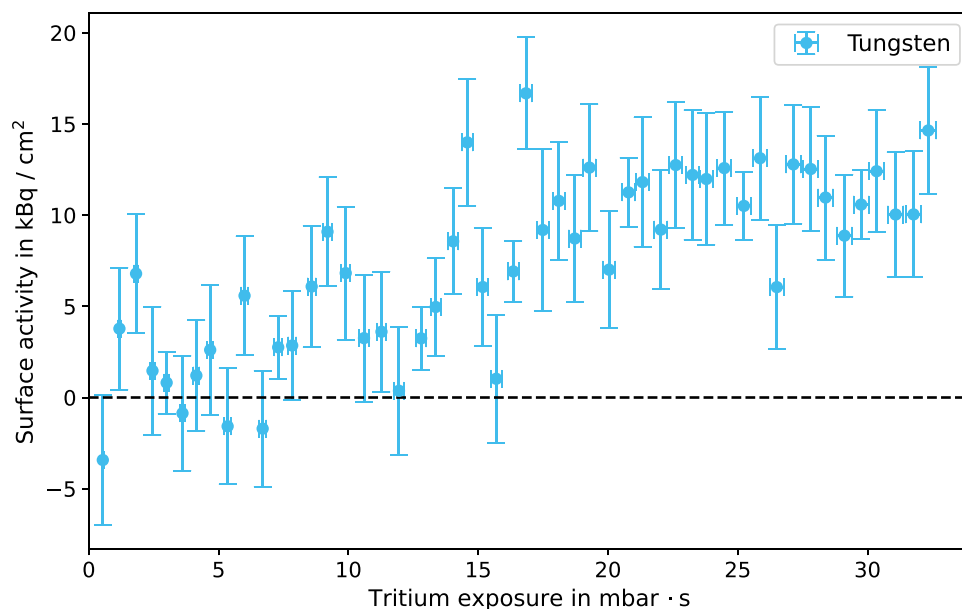


Fig. 3. Surface activity evolution of the tungsten sample.

the impact of ozone, the sample was exposed to dry air taken from the glove box, with and without prior ultraviolet irradiation. In both cases, the air/ozone were kept in the recipient for 20 min to allow the ozone to react with the surfaces and to have a consistent procedure [11]. It is expected that the ozone content in the air was around 200 ppm after the gas mixture was expanded from the ozone generator to the sample chamber. This concentration was measured in another experiment [11] with a similar geometry using Fourier transform infrared spectroscopy. More information on the generation and transport of ozone within a vacuum system is given in Ref. [12]. Similar to the contamination measurements, multiple cycles of ozone and air exposure as well as evacuation were applied.

Fig. 4 shows the influence of the decontamination procedure on the beryllium sample's surface activity, normalized to its starting value. The exposure pressures used for the individual data points are noted in the graph. Breaking the vacuum to connect the ozone generator for the first time was included as the first data point since this already caused the surface activity to decrease by roughly 10%. Two decontamination steps at low pressure using ozone-containing air, two steps with only dry air, and two steps with ozone followed. Each of these was comparable in decontamination efficiency, resulting in an activity of 70% of the initial value after the six steps. The first significant reduction in activity could be achieved after introducing ozone-containing air at a rate of roughly 0.4 mbar/s until a pressure of roughly 800 mbar was reached. A subsequent, rapid exposure at a relatively high

pressure had only a marginal effect on the surface activity. Because of experimental boundary conditions, the decontamination measurements were concluded, with an overall achieved activity reduction of 60%.

The same procedure was applied to the tungsten sample but in a more systematic way, as seen in Fig. 5. After installing the ozone generator, multiple exposures at 50 mbar were carried out, first with dry air without and then with ozone. Each individual step removed up to 3% of the initial surface activity, and no distinction between the absence or presence of ozone could be made. This also applies when higher pressures, in this case, 500 mbar, are used. For the last four measurements, the pressure was slowly increased by 0.2 mbar/s. These did not have any significant decontamination effect, which is different for the beryllium sample. All in all, about a quarter of the initial activity could be removed from the surface of the tungsten sample before finishing the measurements.

IV. DISCUSSION

Each beryllium sample accumulated less tritium on its surface than the previous one. A reason for this could be the fact that most of the components in the vicinity of the sample were in pristine condition before the start of the measurements. As the samples were exposed to tritium, the surrounding surfaces could have been gradually saturated such that their tritium adsorption decreased over time until reaching a stable level. Nevertheless, the

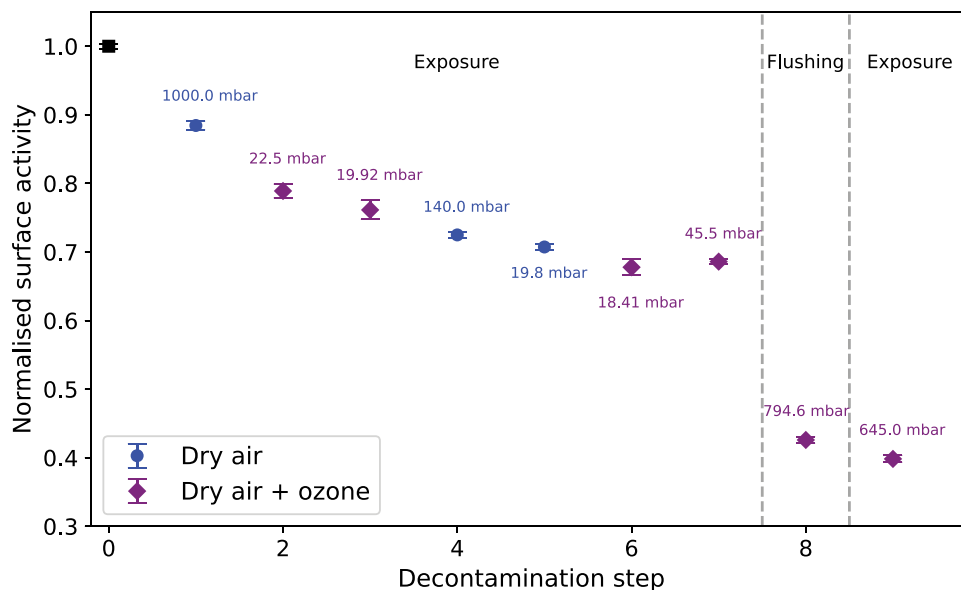


Fig. 4. In situ decontamination of the third beryllium sample with the corresponding pressure during exposure.

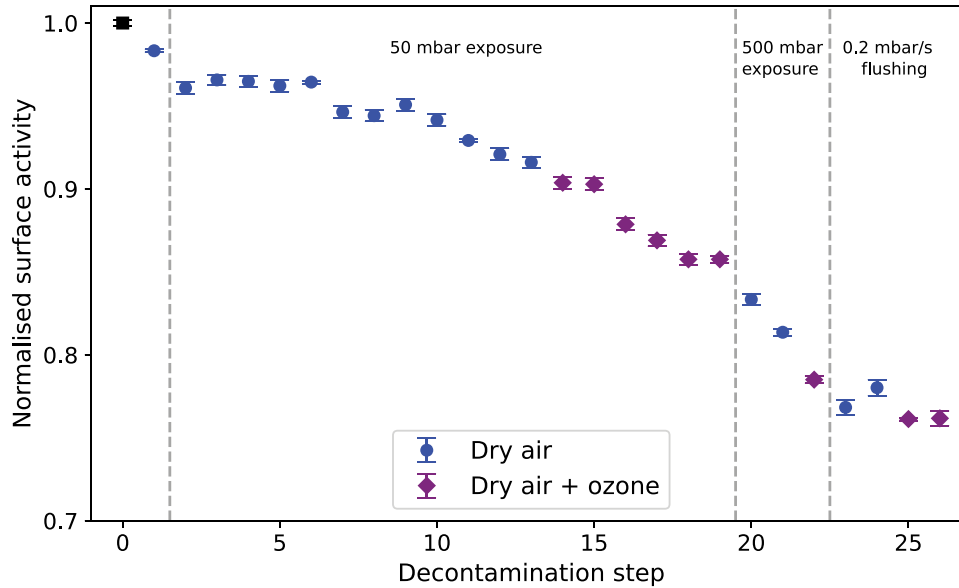


Fig. 5. In situ decontamination of the tungsten sample.

tungsten sample retained significantly less tritium on its surface, namely, less than a quarter compared to beryllium. It is currently unknown which parameters are responsible for this, e.g., different surface properties like structure, area, and defects or the chemical affinity for tritium. These potential differences were not accounted for in this study as the focus was on investigating technical surfaces.

For the decontamination measurements, using dry air with and without ozone reduces the samples' surface activity by comparable amounts. The most probable cause for the tritium desorption is an initial flushing effect at the start of the exposure, for which the ozone is irrelevant.

V. CONCLUSION AND OUTLOOK

With the TRACE setup, it is possible to investigate tritium accumulation as well as decontamination methods using different gases in situ. This was demonstrated using three beryllium samples and a tungsten sample. Assuming that exposure to gaseous tritium in the molecular flow regime is the primary cause of adsorption, beryllium accumulates tritium at a faster rate than tungsten. From the decontamination measurements presented in this work, it is not possible to confirm a beneficial effect of ozone in dry air over dry air alone. If it turns out that simultaneous irradiation with ultraviolet light is indeed required for efficient decontamination, it will potentially be more complicated to carry out in situ decontamination on larger tritiated systems.

Designing the next iteration of the system is currently in progress. The upgraded setup will have the capability to heat up the sample to up to 1500°C, e.g., to reproduce the conditions in a fusion fuel cycle more accurately. Additionally, it will be possible to directly irradiate the sample with ultraviolet light. This way, it will also be possible to investigate the combined in situ ultraviolet/ozone decontamination, which is required to realize and establish the method on a more technical scale.

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Author Contributions

CRedit: **Dominic Batzler:** Conceptualization, Formal analysis, Investigation, Software, Supervision, Visualization, Writing – original draft, Writing – review & editing; **Robin Gröble:** Conceptualization, Supervision, Writing – review & editing; **Philipp Haag:** Conceptualization, Formal analysis, Investigation, Software; **Elizabeth Paine:** Conceptualization, Formal analysis, Investigation, Software; **Marco Röllig:** Conceptualization, Resources, Supervision, Writing – review & editing; **Marie-Christine Schäfer:** Conceptualization, Investigation; **Marius Schaufelberger:** Conceptualization, Formal analysis, Investigation, Writing – review & editing; **Kerstin Trost:** Conceptualization, Formal analysis, Investigation, Writing – review & editing.

Disclosure Statement

No potential conflict of interest was reported by the author(s).

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ORCID

Dominic Batzler  <http://orcid.org/0009-0008-9040-9104>

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