

Hydrogen isotope permeation through interfaces and permeability of tungsten layers

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ABSTRACT

The estimation of the hydrogen isotope flux through a fusion reactor wall component is important for the material selection and in order to guarantee a safe and economical reactor operation. Since the permeation flux through a component cannot be measured directly, due to the large size of such a component, the deuterium permeability of various fusion materials were investigated in the last years. In order to investigate, if an interface, meaning a change of materials in a component, has an influence on the permeation flux, combined material systems in laboratory scale are studied. The combined material systems are produced by applying a thin W or Cu layer on a polished bulk steel or CuCrZr substrates by magnetron sputter deposition. In a previous study (Houben et al., 2022), the combined material system of Cu coated steel was investigated. The conclusion was that the interface has a minor influence on the permeation flux compared to the large influence of the layer microstructure. In order to investigate, if the minor influence of the interface is valid in general, the systems W coated steel and CuCrZr are studied in this publication. By heating the substrate during W sputter deposition, a crack-free W layer is produced and crack propagation in the W layer at elevated temperature is prevented. The W layer permeabilities are obtained for both W coated substrates and are similar, but compared to the W bulk permeability from literature the W layer permeabilities are several orders of magnitude larger.

The main conclusion from these studies is that in all investigated combined material systems the influence of the interface on the permeation flux is minor compared to the large influence of the microstructure. Therefore, for a reliable estimation of the permeation flux through a fusion reactor component it is crucial to characterize the applied materials. Especial for coatings the measurement of the layer permeability is important, since the layer permeability of a material can be very different compared to the permeability of a bulk material.

1. Introduction

The knowledge of the hydrogen isotope permeability of potential fusion reactor wall materials is a crucial factor for the material selection and in order to guarantee a safe and economical reactor operation. For the estimation of the hydrogen isotope permeation flux through a fusion reactor wall component, the deuterium permeability of several steels, copper and the copper alloy CuCrZr-IG (IG:ITER-grade) were investigated in the last years [1,2]. A wall component consist of several materials. Therefore, the question arose, if the interface, meaning the change of the material, e.g. from steel to copper, has an influence on the permeation flux. If such an influence is measurable, the permeation flux through a component cannot be estimated by using the permeability of the single materials only. Since a direct permeation flux measurement

through a complete component is not possible due to the size of the component, studies are performed on combined material systems in laboratory scale. These laboratory scaled combined material systems are produced by depositing thin Cu and W layers by magnetron sputter deposition on polished steel and CuCrZr-IG substrates. By measuring the deuterium permeability of the combined systems in comparison to the single materials, the influence of the interface is experimentally determined. A theoretical study on the same aspect can be found in [3].

The first studied combined material system was the Cu coated 316L(N)-IG steel system and the results are published in [2]. In this study, it was noticed that the microstructure of the layer is much different compared to an industrial produced Cu (bulk) sample. The

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bulk Cu sample showed a much larger grain size and voids compared to the dense magnetron sputter deposited Cu layer. Since the microstructure can have a large influence on the permeation flux [4,5] and a permeation measurement through a single Cu layer is not possible, three Cu layered steel substrates with different layer thicknesses were studied. Due to the different layer thicknesses, the layer bulk and interface ratio was varied. By varying the ratio, the effect of the interface and microstructure on the permeability can be separated. The conclusion from the study was that the influence of the interface on the permeation flux was minor compared to the large influence of the layer microstructure on the permeation flux. The Cu layer permeability is more than one order of magnitude smaller compared to the bulk Cu sample.

In order to investigate, if the minor influence of the interface is in general valid also for other combined material systems, W layers are deposited on polished Eurofer97 (named Eu97) and CuCrZr-IG (named CuCrZr) substrates and the results are reported herein. The W layer microstructure and thickness are kept constant and the substrates are polished before the deposition. The deuterium permeability of the two used substrates are different by two orders of magnitude in the measured temperature range [1,2]. Due to the large difference in the substrate permeability, the comparison of the permeability of the combined material systems by the commonly used permeation reduction factor (PRF) is not possible. Therefore, next to the investigation of the influence of the interface on the permeation flux, the reliability of the calculated layer permeability [6] will be shown.

The thermal expansion coefficient of W ($4 \cdot 10^{-6}$ 1/K) is much lower than the thermal expansion coefficients of steel and CuCrZr ($12 \cdot 10^{-6}$ 1/K and $17 \cdot 10^{-6}$ 1/K, respectively). That is why stress is induced in the W layer if the W coated substrates are heated. This stress leads to cracks and crack propagation in the W layer during annealing and permeation measurements, which are performed up to 550 °C, typically. It was noticed in a former study [7] on W coated steel substrates that cracks in the W layer acts as shortcuts to the substrates for the hydrogen and this leads to an increase of the permeation flux. Furthermore, due to crack propagation during the permeation measurement, no stabilized permeation flux measurement was possible. In the present study, stable W layers are obtained and by comparison of cracked and non-cracked W layers, the influence of cracks and crack propagation on the permeation flux measurement will be discussed.

2. Sample preparation

The Eu97 and CuCrZr substrates are grinded and polished according to standard procedures described in detail in [1,2]. After polishing, the disk shaped substrates (diameter: 24 mm) are around 0.4 mm thick.

For the deposition of the W layer on one side of the substrate, a magnetron sputter deposition device (PREVAC) is used. The deposition is performed with a pure W target (99.95% W, Lesker) and an Ar plasma (DC, 300 W). The base pressure in the magnetron deposition device is 10^{-7} mbar before deposition. The high vacuum prevents the formation of the oxygen stabilized β -W phase. During deposition, the sample stage is rotated in order to obtain a homogeneous layer.

In a first attempt to avoid the W layer cracking at elevated temperatures due to the different thermal expansion coefficients, a 100 nm thin W layer is deposited on a CuCrZr substrate and the maximum applied temperature during pre-annealing and permeation measurement is reduced to 450 °C. This attempt failed, because already during pre-annealing cracks appear and crack propagation are observed during permeation measurement. This sample will be named CuCrZr_W-cracked in the following and the results will be used for the discussion of the influence of cracks, only. In a second attempt to obtain a crack free W layer, a substrate heating is applied during the W deposition. The substrates are heated to around 300 °C during deposition in order to shift the temperature range, in which the layer stays crack free, to higher temperatures. This attempt is successful, the

occurrence of cracks in the layer is reduced, and a crack propagation in the measurement range up to 550 °C is prevented.

In order to have several identical samples for the different measurement methods, three Eu97 substrates are deposited at the same time in one deposition process. The thickness of the W layer on the Eu97 substrates is about 330 nm and the samples from this batch will be named Eu97_W in the following. In a second W deposition process, two CuCrZr substrates are prepared at the same time, the W layer thickness is about 350 nm and the samples from this batch will be named CuCrZr_W. Typically, the substrate has no influence on the microstructure of magnetron deposited layers, therefore identical W layer microstructures are expected on both substrates, since the deposition parameter are kept constant.

After deposition and before the permeation measurement, all samples are pre-annealed under vacuum at the maximum applied temperature of 550 °C for several hours. With this procedure, the native hydrogen content is removed from the sample up to this temperature and a stable sample condition (no cracks in the coating, no peeling of the coating etc.) is proven.

3. Measurement methods

After the W layer deposition and after the pre-annealing, the W crystal phase is obtained by X-ray diffraction (XRD, Bruker, D8 Discover, Cu tube) on all samples. The surface of the coatings is analyzed by scanning electron microscopy (SEM) with a Crossbeam 540 device (Zeiss) after pre-annealing and before permeation measurement in order to verify, if cracks in the layer appear due to the thermal treatment. After the permeation measurement, the surface and the cross sections, which are prepared by a focused ion beam (FIB), are studied by SEM in the same device. Furthermore, the layer thicknesses are measured on the cross sections.

The gas-driven permeation flux measurements are performed in the in-house permeation setup with the standard procedure [2] and in the sample temperature range between 300 °C and 550 °C and between 25 mbar to 800 mbar applied deuterium pressure range. The time between two pressure steps is adjusted to the sample permeability and a stabilization of the permeation flux at each pressure step is ensured. After performing the “up-measurements” (300 °C, 400 °C, 500 °C, 550 °C), the “down-measurements” are performed at decreasing temperatures (500 °C, 400 °C, 300 °C). With this procedure, a change of the sample state can be identified. If the corresponding up- and down-measurements are similar, a change of the sample state during permeation measurement can be excluded. The permeation flux is measured by a quadrupole mass spectrometer (Pfeiffer Vacuum) and the signal is calibrated by four D₂ calibration leaks (LACO) with different fixed deuterium fluxes. The base pressures of the high pressure volume (HPV, applied deuterium side) and low pressure volume (LPV, mass spectrometer side) are in the 10^{-9} mbar and 10^{-10} mbar range, respectively. After the permeation measurement, the thicknesses of the coated substrates are measured by a micrometer screw.

4. Data analysis

The stabilized deuterium permeation flux is measured in the temperature and pressure range indicated above on the Eu97_W and the CuCrZr_W samples. From the dependence on the applied deuterium pressure, the process limiting regime can be determined: If J_p is proportional to the square root of the applied pressure, diffusion is limiting the process. In the diffusion limited regime, J_p is dependent on the sample or layer thickness and the permeability constant P_0 and the activation energy E_p can be obtained:

$$J_p = \frac{P_0 \sqrt{p}}{d} e^{\frac{-E_p}{RT}} \quad (1)$$

wherein d is the thickness of the sample, R is the ideal gas constant and T the sample temperature. The obtained permeability $P = P_0 e^{\frac{-E_p}{RT}}$ is

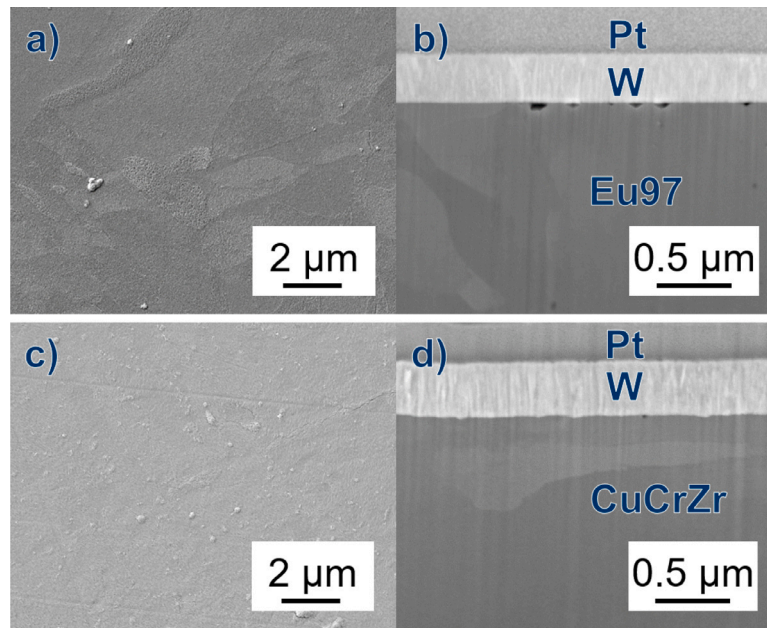


Fig. 1. SEM images of the surfaces (a, c) and FIB prepared cross sections (b, d) of: (a)–(b) Eu97_W after permeation measurement; (c)–(d) CuCrZr_W after pre-annealing. In the FIB prepared cross sections, the materials are indicated. On top of the samples, a Pt layer is applied before cutting in order to avoid surface damage and to improve cross section quality during the preparation process.

valid in the measured temperature and pressure range only. If surface effects are slower as the diffusion and therefore limiting the process, the permeation flux J_p is linear dependent on the applied pressure p . Furthermore, if interface effects influences and determining the permeation process, a deviation from Eq. (1) is expected and the thickness and pressure dependence is not given any more.

The layer permeability P_{lay} can be calculated by the permeability of the substrate P_{sub} and the permeability of the coated substrate P_{tot} :

$$P_{lay} = \frac{d_{lay}}{\frac{d_{tot}}{P_{tot}} - \frac{d_{sub}}{P_{sub}}} \quad (2)$$

wherein d_{tot} , d_{sub} , and d_{lay} are the thicknesses of the layered substrate, the substrate and the layer, respectively [6]. Eq. (2) is valid in the diffusion-limited regime, only. Nevertheless, we will use the layer permeability, if the permeation process is not strictly diffusion limited, as well. In this case, the layer permeability is an estimation after deduction of the substrate permeability from the total system permeability. It contains all effects which influence the permeability such as surface, interfaces, microstructure and further properties of the layer. With this calculated layer permeability, the comparison of layers on substrates with different permeability is enabled, which would be impossible by the PRF value only, which is defined by the permeation flux of the bare substrate divided by the permeation flux of a coated substrate. In the following, if the values refer to the W layer only, i.e. after deduction of the substrate, the names $W_{layer(Eu97)}$ and $W_{layer(CuCrZr)}$ will be used for the Eu97_W and CuCrZr_W samples, respectively.

5. Results

The crystal phase analysis by XRD identify the cubic (bcc) W crystal structure in all layers, as expected (not shown).

In Fig. 1, the surface (a) and cross section (b) SEM pictures of the Eu97_W sample after the permeation measurement are shown. No cracks and a clean interface without intermediate phases are observed. The small voids in the substrate are due to the polishing procedure of the substrate and have no influence on the W layer. The obtained

thickness of the W layer is 330 nm. For the CuCrZr_W sample, the same SEM pictures are shown after pre-annealing (Fig. 1c-d). These figures confirm that the microstructure of the W layer and the interface in the CuCrZr_W sample are similar compared to the layer in the Eu97_W sample and the thickness of the tungsten layer of 350 nm is measured. In Fig. 2 left, a surface SEM picture of the CuCrZr_W sample after permeation measurement is shown. In opposite to the Eu97_W sample, large, very straight cracks are observed after the permeation measurement. The origin for the crack formation and the influence on the permeation flux measurements will be discussed in Section 6. On the right side of Fig. 2, a surface SEM figure on the W layered CuCrZr sample without substrate heating during deposition (CuCrZr_W_cracked) is shown for comparison.

As an example, the raw permeation flux measurement data at 500 °C for the Eu97_W and CuCrZr_W samples are shown in Fig. 3. The differently dotted black lines indicate the stabilized permeation flux at the different applied pressures. The values are obtained by fitting a constant in the stable flux area. Furthermore, it can be observed that a stable permeation flux is reached within seconds. The slightly larger increase time at 400 mbar and 800 mbar are due to the reason that the pressure increase in the HPV takes some minutes to reach the next pressure step. No change of sample state is observed from the comparison of the up- and down measurements in both samples.

In Fig. 4 the Arrhenius plot for the Eu97_W samples is shown as an example. The activation energy is obtained from the slope of each fitted line and the permeation constant is obtained from the intercept according to Eq. (1) for all pressures. In Table 1 the mean values for E_p and P_0 are given for both samples. Furthermore, the pressure dependence is obtained from the stable permeation flux data and given in Table 1. For comparison, the values for the uncoated substrates are given as well and the permeability for the four samples in the measured temperature range is shown in Fig. 5.

The layer permeabilities of all W layers are calculated according to Eq. (2). For the W layer permeability of the Eu97_W system ($W_{layer(Eu97)}$), the Eu97 value is used for the substrate permeability and for the layered substrate permeability the values for Eu97_W are taken

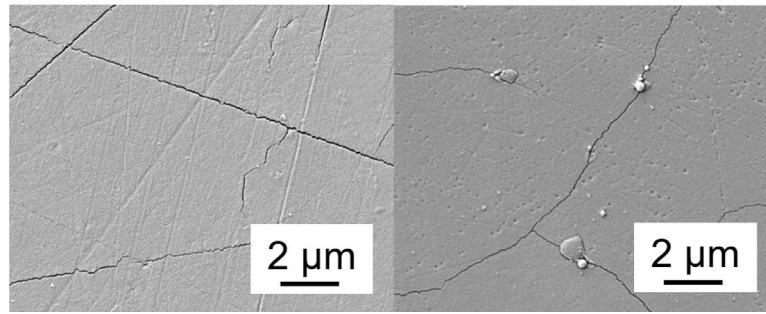


Fig. 2. Surface SEM of CuCrZr_W after permeation measurement up to 550 °C (left) and the surface SEM CuCrZr_W-cracked sample after annealing at 450 °C (right).

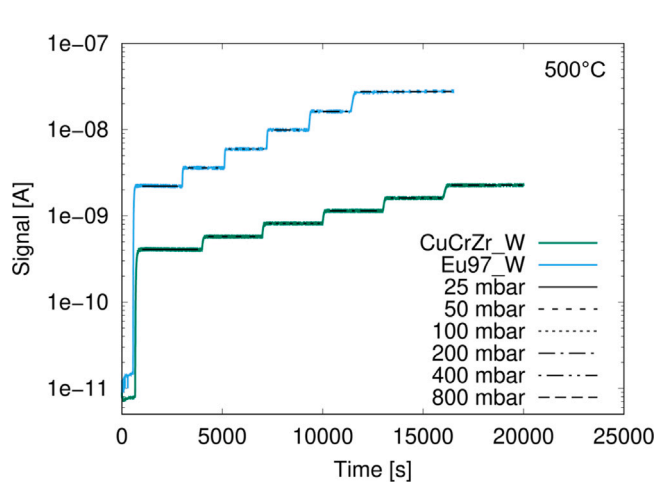


Fig. 3. Mass 4 signal for the Eu97_W sample (blue) and CuCrZr_W sample (green) at the permeation flux measurement at 500 °C. The black lines indicate the fitted stable flux values for the different applied deuterium pressure steps. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

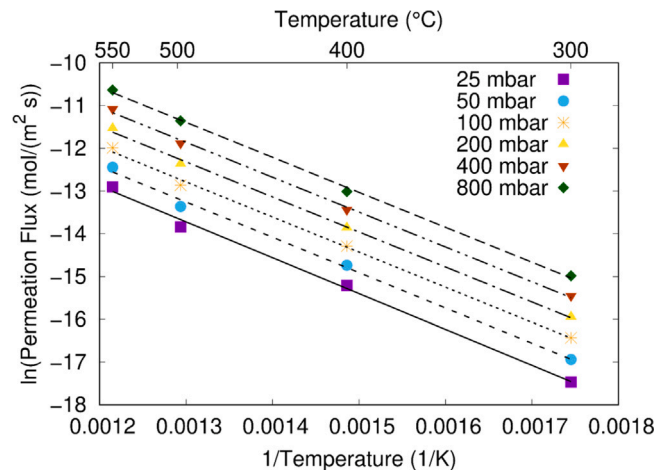


Fig. 4. Arrhenius plot for the Eu97_W sample. The different symbols indicate the different pressure steps and the black lines the corresponding fits.

from Table 1. For the W layer permeability of the CuCrZr_W system ($W_{\text{layer}(\text{CuCrZr})}$), the CuCrZr and CuCrZr_W values are used, respectively. The layer permeabilities can be found in Table 2 and are plotted in

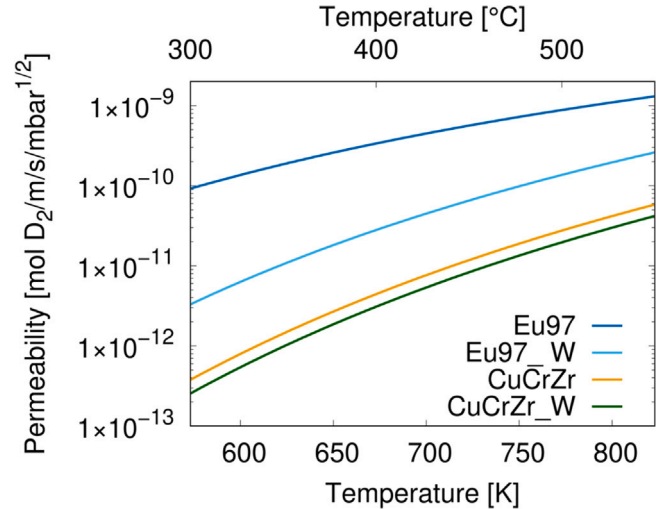


Fig. 5. Comparison of the permeabilities of the coated systems Eu97_W (bright blue) and CuCrZr_W (green) with the uncoated substrates Eu97 (blue) and CuCrZr (orange) from Table 1. The shown temperature range in K (lower x axis) and °C (upper x axis) corresponds to the measured temperature range. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 1

The results obtained from temperature and pressure dependent permeation measurements: pressure dependence p^* , permeation constant P_0 , and activation energy E_p . The values for the Eu97 substrate are taken from [1] and for the CuCrZr from [2].

Sample	p^*	P_0 [$\frac{\text{mol}}{\text{ms}\sqrt{\text{mbar}}}$]	E_p [$\frac{\text{kJ}}{\text{mol}}$]
Eu97_W	0.65	$(5.8 \pm 0.5) \cdot 10^{-6}$	68.5 ± 0.5
CuCrZr_W	0.5	$(5 \pm 1) \cdot 10^{-6}$	80 ± 1
Eu97[1]	0.5	$(5.7 \pm 0.4) \cdot 10^{-7}$	41.6 ± 0.5
CuCrZr [2]	0.55	$(6 \pm 2) \cdot 10^{-6}$	79 ± 1

Fig. 6. For comparison, the for deuterium adapted W bulk permeability from literature [8] is shown in Table 2 and plotted in Fig. 7.

6. Discussion

The comparison of the permeabilities of W coated and uncoated Eu97 and CuCrZr is shown in Fig. 5. The large difference of the permeabilities of the two substrate materials Eu97 (blue) and CuCrZr (orange) is clearly visible and is about two orders of magnitude. Due to W coating, a reduction of the permeability is observed in both cases Eu97_W (bright blue) and CuCrZr_W (green), as it is expected, since W shows a lower permeability compared to Eu97 and CuCrZr in the

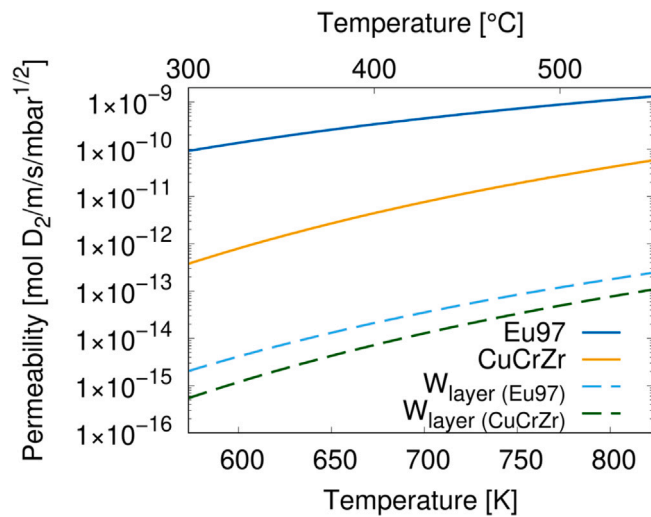


Fig. 6. Comparison of the W layer permeabilities (dotted lines) $W_{\text{layer(Eu97)}}$ (bright blue) and $W_{\text{layer(CuCrZr)}}$ (green) with the uncoated substrates Eu97 (blue) and CuCrZr (orange). The layer permeabilities are calculated from the permeabilities of the Eu97_W and CuCrZr_W samples by deduction of the substrate permeability, see Table 2. (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

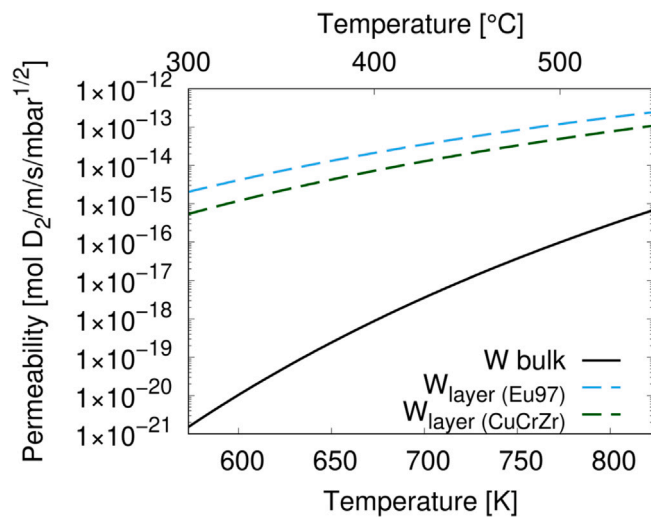


Fig. 7. Comparison of the W layer permeabilities $W_{\text{layer(Eu97)}}$ (bright blue) and $W_{\text{layer(CuCrZr)}}$ (green) with the W bulk values (black). The values for the W bulk permeability are taken from [8] and adapted to deuterium. Please note that the W bulk permeability was obtained in a different temperature range (between 1100 K and 2400 K). (For interpretation of the references to color in this figure legend, the reader is referred to the web version of this article.)

Table 2

The layer permeabilities calculated according Eq. (2). The values for the W bulk permeability are taken from [8] and adapted to deuterium. Please note that the W bulk permeability was obtained in a different temperature range (between 1100 K and 2400 K).

Layer	P_0 $\left[\frac{\text{mol}}{\text{ms}\sqrt{\text{mbar}}} \right]$	E_p $\left[\frac{\text{kJ}}{\text{mol}} \right]$
$W_{\text{layer(Eu97)}}$	$1.4 \cdot 10^{-8}$	75
$W_{\text{layer(CuCrZr)}}$	$2 \cdot 10^{-8}$	83
W bulk [8]	$6 \cdot 10^{-3}$	204

measured temperature range. The reduction in the Eu97_W sample due to the W layer is obviously much larger compared to the CuCrZr_W sample. This is due to the lower permeability of CuCrZr. Due to this fact, a reliable comparison of the coating via the PRF value is not possible and the PRF value for an identical W layer in the Eu97_W sample would be around one magnitude larger as in the CuCrZr_W sample. As it is shown in Fig. 6, a comparison of the calculated W layer permeabilities $W_{\text{layer(Eu97)}}$ and $W_{\text{layer(CuCrZr)}}$ obtained from the Eu97_W and CuCrZr_W samples is possible and the W layer permeability is in the same order of magnitude. Since the W layer permeabilities are similar and the pressure dependence shows a more diffusion limited permeation process (Table 1), it is concluded that the influence of the interface on the permeation flux is minor, as it was observed in the Cu coated steel system [2] as well. We want to point out that this conclusion is valid for a clean interface only. If there would be an intermediate phase between the two materials, e.g. oxides, as it is expected in real components, the intermediate phase can have an influence on the permeation flux. In [1] we studied the influence of oxidized and rough surfaces on the permeability. The conclusion of this study was that the permeability can be lowered by maximal one order of magnitude due to a technical surface.

From the comparison on the W layer permeabilities and the W bulk permeability, see Fig. 7, it can be concluded that the influence of the microstructure is large. In the Cu coated steel system from the previous study [2], the same conclusion was drawn from the results, but in opposite to the W layer permeability, the Cu layer permeability was smaller as the Cu bulk permeability. This is a strong indication that the microstructure is a crucial factor for the permeation flux and has to be considered for the use of a material in a fusion device. In order to calculate the permeation flux through a fusion component, the applied materials have to be pre-characterized, e.g. by SEM and permeation measurements on the used materials. This is especially important for coatings and the measurement and calculation of the layer permeability enables a reliable estimation of the permeation flux through a fusion component.

For the discussion of the influence of cracks on the permeation flux, the surface SEM figure of the CuCrZr_W sample after the permeation measurement is shown in Fig. 2 left. Large, very straight cracks can be observed, which were not observed before the permeation measurement (Fig. 1c). On the right side of Fig. 2, the surface image of the W coated CuCrZr sample from the first, failed attempt of crack-free W layer deposition (CuCrZr_W_cracked) is shown. The 100 nm thick W layer was deposited without substrate heating and showed cracks after the pre-annealing at 450 °C already. By SEM on a cross section (not shown) it was observed, that in both cases the cracks are through the W layer completely. Comparing the surface SEM figures, a different shape of the cracks can be seen, straight in the CuCrZr_W sample on the left side of Fig. 2 and curved in the CuCrZr_W_cracked sample (right). The cracks in the CuCrZr_W_cracked layer are due to the stress induced by the different thermal expansion coefficient of CuCrZr and W. Furthermore, no change of permeation flux during permeation measurement is observed in the CuCrZr_W sample and a stable permeation flux measurement is possible, see Fig. 3. In the case of crack propagation during the permeation flux measurement, a non-stable permeation flux measurement was observed in the previous study on W coated steel [7]. Therefore, no change of the CuCrZr_W sample state, e.g. crack propagation, is expected during the permeation measurement. Furthermore, due to the different appearance of cracks in the W layer it is concluded that the reason for the cracks in the CuCrZr_W sample is not due to the thermal induced stress, but is due to mechanical treatment. The assumption is that the cracks appear during sample installation, since the sample is clamped between two gasket rings in the permeation device and the sample is slightly bended due to the softness of the CuCrZr substrate and the force of the clamping. The conclusion is that these static cracks do not have a measurable influence on the permeation flux in opposite to the crack propagation in the W layer in reference [7].

7. Conclusions

Two different substrates, Eu97 and CuCrZr, are coated with W by magnetron sputter deposition. By heating the substrates to 300 °C during deposition, crack-free W layers up to the maximum applied temperature of 550 °C are produced. On these combined material systems, deuterium gas-driven permeation measurements are obtained and the W layer permeability was calculated. The W layer permeabilities of the Eu97_W sample and the CuCrZr_W are similar and more diffusion limited, which verify that the influence of the interface is minor and that a reliable comparison of layers applied on different substrates is possible with the calculated layer permeability. Compared to a W bulk sample, the W layer permeabilities are several orders of magnitude larger, indicating the large influence of the microstructure on the permeation flux. Furthermore, it is observed that static cracks in a layer due to mechanical treatment have no measurable influence on the permeation flux, whereas cracks and crack propagation, which appear due to the stress induced by different thermal expansion coefficient at elevated temperatures, have an influence on the permeation flux [7].

The main conclusion obtained from the studies on combined material systems is that the influence of interfaces is minor compared to the large influence of the microstructure on the permeability. For a reliable estimation of the hydrogen isotope permeation flux through a fusion component, the applied materials have to be carefully characterized. This is especially important for coatings, since the layer permeability can be much smaller or larger as the permeability of a bulk material.

CRediT authorship contribution statement

A. Houben: Conceptualization, Methodology, Formal analysis, Investigation, Writing – original draft, Visualization, Project administration. **M. Rasiński:** Formal analysis, Investigation. **S. Brezinsek:** Conceptualization, Resources, Writing – review & editing. **Ch. Linsmeier:** Conceptualization, Resources, Writing – review & editing, Funding acquisition.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Anne Houben reports financial support was provided by Eurofusion European Consortium for the Development of Fusion Energy.

Data availability

Data will be made available on request.

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