Sticking coefficient and surface loss probability of eroded species during bombardment of carbon with deuterium

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Abstract

Investigations on the deposition behaviour of eroded carbon species under controlled ion beam conditions were performed to evaluate absolute sticking coefficients. Pyrolytic carbon was eroded by D ions at energies between 45 eV and 1 keV and temperatures between 20 and 475 °C. The sticking coefficient of the eroded species was obtained from a quantitative comparison of the total deposition inside a cavity with the flux of carbon entering the cavity as well as from the distribution of deposited atoms inside the cavity. High sticking coefficients of the order of 0.6 were obtained at room temperature and high ion energy, as expected from physically eroded atoms. In contrast, hydrocarbon molecules chemically emitted at either elevated temperature or low ion energy have typical sticking coefficients around 0.02. At elevated temperatures, the quantitative comparison with weight-loss measurements indicate the dominance of saturated hydrocarbons with zero sticking to the cavity walls.
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1. Introduction

The use of carbon tiles at the first wall of fusion devices is challenged by the large amounts of hydrogen isotopes retained in layers of carbon atoms deposited after erosion. Extrapolations from present experiments indicate that within few plasma discharges the site limit for tritium may be retained in these layers [1]. Investigations in fusion devices [2,3] have revealed a complicated deposition sequence involving the release of hydrocarbon radicals from the divertor plates which finally accumulate on cool remote surfaces.

The nature of the released hydrocarbons and, therefore, their sticking coefficient to surfaces, depend critically on local plasma and surface properties. It is known that different hydrogen ion energies and surface temperatures result in different erosion regimes of carbon [4]:

- At room temperature and energies in the keV range, predominantly physical sputtering occurs with carbon atoms being eroded.
- At elevated temperatures, chemical erosion increases the erosion yield to a maximum around 500 °C, with methane being the dominant emitted hydrocarbon, but with important contributions from heavier hydrocarbons increasing with decreasing ion energy [5].
- Another process occurs at low energies, i.e. below 100 eV where physical sputtering is negligible, and room temperature [6,7]. Due to bond-breaking events and passivation of the resulting dangling bonds by the incident hydrogen species volatile hydrocarbons are formed at and below the surface [8–10]. In this case a complex mixture of saturated hydrocarbons and hydrocarbon radicals is expected to be released.

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The sticking coefficient to surfaces and the surface loss probability of the emitted species is of high importance for the understanding of the carbon deposition and transport in fusion devices. Most favourable would be the transformation of hydrocarbon radicals into saturated hydrocarbons with negligible sticking to walls, thereby preventing the accumulation of large amounts of tritium inside the fusion vessel and enabling effective pumping of hydrocarbons. In the present investigation, the surface loss probability of the erosion products resulting from the three erosion processes outlined above will be directly determined using the cavity technique. Furthermore, quantitative conclusions on the sticking coefficient are drawn based upon an estimation of the carbon flux towards the cavity probe.

2. Cavity technique and data analysis

The surface loss probabilities were determined employing the cavity technique which was previously described in detail [11–13]. Its basic principle is that a flux of reactive particles entering a defined geometry through a slit deposits a film on the inside surfaces. The spatial variation of the film thickness is then determined by the surface loss probability β, i.e. the probability that the particle does not survive a wall collision as the initial species; it either sticks to the wall (probability s) or reacts to a volatile, non-reactive species (probability γ), in other words $\beta = s + \gamma$. Thus, the surface loss probability is an upper limit for the sticking coefficient, $s \leqslant \beta < 1$.

It is important to emphasise that the shape of the film thickness profile solely depends on β. For the geometry used in this work the technique is reasonably sensitive to species with surface loss probabilities between 1 and 10^{-2} whereas any smaller values of β result in almost indistinguishable homogeneous deposition profiles on the inner surfaces. The film thickness profiles are evaluated by comparing the measured profiles to model calculations for molecules with different surface loss probabilities. For most of the profiles more than just one incoming species have to be assumed. More details concerning the experimental set-up and the evaluation calculations can be found in [12,13].

Fig. 1 shows a cross-sectional view of the set-up. The graphite target was placed in the centre of a cylindrical stainless-steel Faraday shield with an inner diameter of 34 mm and a height of 90 mm. The ion beam enters through a hole in the cylinder wall. The cavity was placed with the entrance slit at 40° to the incident beam direction as seen in Fig. 1. The inner height of the cavity is 2 mm, the side walls are 14.5 mm apart and the entrance slit is 0.8 mm wide and 8.5 mm long in the direction perpendicular to the depicted cross-section. Both, the bottom and top surfaces are made from 0.4 mm thick silicon wafers.
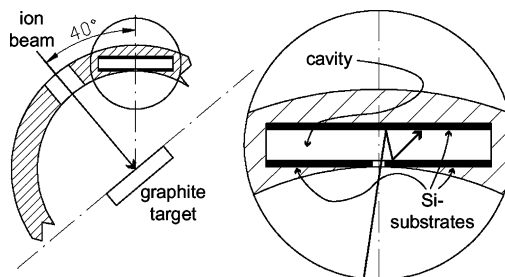

Fig. 1. Schematic arrangement of sputter target and collector cavity.

The physically sputtered and chemically eroded particles as well as reflected deuterium or helium reached the cavity probe and created a measurable carbon layer. After each experiment the cavity was disassembled and the deposited films on the inner Si surfaces were analysed by MeV ion beam techniques. The deposited carbon was detected with 1.735 MeV proton enhanced backscattering (PES) at a scattering angle of 165°. The sensitivity of this method is high enough to assess the natural coverage of the surface of the Si wavers to below 10^{15} cm^{-2}. A further advantage of the method is that it directly yields the carbon areal density. Hence, in contrast to film thickness measurements the result is not influenced by potential variations of the carbon atom density of the films. Therefore, in the remainder of this article we speak about the '(spatial) distribution of carbon' in the cavities rather than the 'film thickness profile'.

The cross-sectional distribution of carbon was measured on the inner Si surfaces along a central line perpendicular to the slit. The total amount of carbon was estimated from integration over the inner Si surfaces assuming that the film thickness does not vary in the direction parallel to the slit. Deposition on the lateral boundaries of the cavities was not taken into account for the following reasons: (1) The side walls perpendicular to the slit are neglected in the simulations of the deposition inside the cavity which are performed in two dimensions and, hence, correct only for an infinitely long slit. Thus, for comparison the experimental distributions have to neglect these side walls, too. (2) The contribution of the cavity walls parallel to the slit is only important for homogeneous deposition inside the cavity as otherwise the deposition strongly decreases laterally. The upper limit of this error for homogeneous deposition is given by the ratio of wall height and wall distance to about 14%.

As pointed out above, the shape of the distribution of carbon in the cavity contains information only on the surface loss probability and is reasonably sensitive only for $\beta > 10^{-2}$. However, a lower limit to the surface loss probability can be given also for the low-β species based

upon a quantitative comparison of the carbon fluence Φ entering the cavity with the amount of carbon retained in the cavity Σ: the probability $p_{esc}(\beta)$ of particles to escape again through the entrance slit (see Fig. 2) after having entered the cavity grows with decreasing β and is almost unity for $\beta = 10^{-4}$. Thus, assuming $s = \beta$, the surface loss probability β_{min} can be derived by

$$\Sigma = \Phi(1 - p(\beta_{min})). \tag{1}$$

For $s < \beta_{min}$ the surface loss probability has to be higher than β_{min} as only the fraction s/β of the non-escaping particles is deposited and, hence, less particles may escape the cavity. Furthermore, it can also be shown that for $\beta > \beta_{min}$ the sticking probability is always higher than β_{min}. Thus, the value β_{min} is a lower limit to both the surface loss probability and the sticking coefficient, or $\beta_{min} < s < \beta$.

The distance between the entrance slit of the cavity and the centre of the beam spot on the target is 17 mm, resulting in a solid angle of 0.023 sr for the aperture slit. Assuming a cosine distribution of the ejected species the fraction directly entering the slit amounts to 5.3×10^{-3} for normal incidence and 7.5×10^{-3} for an angle of incidence of 30° of the ion beam with respect to the surface normal of the graphite target. For low surface loss probability the particles can, however, also enter the slit after several collisions with the walls of the cylinder. The resulting flux of particles to the slit eventually becomes comparable to the direct flux from the target. We have estimated the indirect flux by means of a simple Monte Carlo simulation: for $\beta = 0.6$ the indirect flux into the slit is generally not much larger than 10% of the direct flux. In contrast, for $\beta = 0.02$ or smaller the indirect flux is of the same order as the direct flux. The total

fluence Φ of species with low β will therefore be by a factor of two or three higher than the direct flux.

3. Experimental methods

The erosion measurements were performed at the Garching high current ion source [14]. The source was adjusted to produce mass separated ion beams of D_3^+ or He^+ in the energy range of 1–5 keV. These ions were slowed down to the chosen impact energy by biasing the target. For all data presented here the ion beam hit the surface either perpendicularly (angle of incidence = 0°) or at 30°. In the latter case the angle between the surface normal of the target and the direction towards the slit is 10°. The bombardment spot had sizes between 0.3 and 0.5 cm² depending on the impact energy. The ion current, as measured using a double Faraday-cup around the target, was about 10 μA for D_3^+ and 2.5 μA for He^+ ions, corresponding to flux densities of about 6×10^{18} D/m² s and 5×10^{17} He/m² s.

The total erosion yield was determined by measuring the weight loss ex situ with a Mettler microbalance (sensitivity ±1 μg) and the total ion charge. For measurements at elevated temperatures the targets were mounted on a plate heated by a thermo-coax. The temperature was determined by a thermocouple.

For the present data pyrolytic graphite from Union Carbide with the surface parallel to the graphitic planes was used as targets.

4. Results and discussion

The erosion experiments were performed under four different conditions: 3 keV He^+ bombardment, 1 keV D^+ bombardment at room temperature (both representative of physical sputtering), 1 keV D^+ bombardment at 475 °C (representative of thermal chemical erosion) and 45 eV D^+ bombardment at room temperature (representative of chemical sputtering). In all four cases the weight loss of the graphite target, the total amount of carbon and the distribution of carbon in the cavities were evaluated.

4.1. Weight loss and sputtering yield

The ion dose, weight loss and erosion yield for the different conditions are given in Table 1. For comparison also the number of eroded atoms directly entering the cavity, Φ, is given under the assumption of a cosine distribution of emitted particles as explained in Section 2. For near normal incidence of bombarding ions this assumption is fairly correct for physical sputtering [15] and can reasonably be assumed for thermal emission of volatile molecules. Also given is the total number of
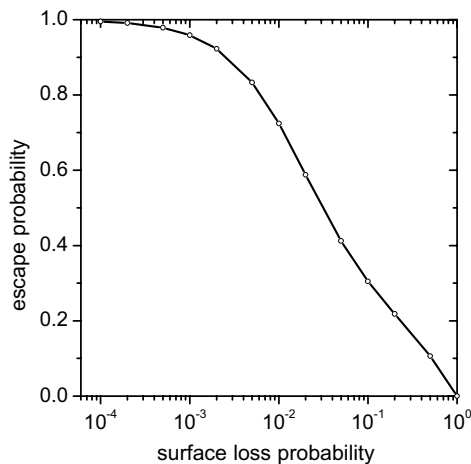

Fig. 2. Probability of a particle to escape through the slit after having entered the cavity as a function of the surface loss probability.

Table 1
Parameters of the four cavity experiments

	Experimental parameters			
	3 keV He$^+$, RT	1 keV D$^+$, RT	1 keV D$^+$, 475 °C	45 eV D$^+$, RT
Regime	Physical sputtering	Physical sputtering	Thermal chemical erosion	Chemical sputtering
Angle of incidence	0°	30°	30°	0°
Ion dose (atoms)	4.1×10^{18}	4.3×10^{19}	1.4×10^{19}	2.4×10^{19}
Weight loss (µg)	n.a.	30.9	60.9	21.1
Sputtering yield	6×10^{-2} [5]	3.6×10^{-2}	2.2×10^{-1}	4.4×10^{-2}
C atoms found in cavity, Σ	1.22×10^{15}	7.91×10^{15}	8.5×10^{15}	3.1×10^{15}
Particles directly entering cavity, Φ	1.3×10^{15}	1.2×10^{16}	2.3×10^{16}	5.6×10^{15}

carbon atoms found within the cavity, Σ, integrating the carbon surface coverage over all inner Si surfaces as described in Section 2.

The total sputtering yields determined from the weight loss of the samples agree well with previously published data for similar conditions and in situ vacuum balance measurements [16]. The comparison of the total amount of carbon found inside the cavity with the number of particles estimated to enter the slit already gives first indications about the sticking coefficient. For physical sputtering, i.e. sputtering at room temperature in the keV energy range, almost all entering atoms are found within the cavity indicating a sticking coefficient close to unity. For the erosion process at room temperature, but at energies close to the threshold energy for physical sputtering also considerable retention within the cavity is found, in agreement with previous open collector measurements [16], which resulted in a sticking coefficient close to 0.5. In the case of a mixture of physical sputtering and thermal chemical erosion, i.e. at 475 °C and 1 keV, the retention is clearly lower. With the assumption that the physically sputtered atoms have a similarly high sticking as at room temperature, the dominantly eroded hydrocarbon molecules appear to have much lower sticking to the collector surface. The comparison of the uniformly distributed fraction within the cavity with the number of particles directly entering the cavity results in a release of 60%. A release of 60% occurs already at a sticking coefficient of 0.02 (see Fig. 2) [12,13]. Taking into account that for low-β species the total incident flux through the slit may be by a factor of two or three higher than the direct flux, the lower limit of the sticking coefficient decreases to about 0.01.

4.2. Distribution of carbon in the cavities

The evaluation of the global retention within the cavity can only give information on the average sticking coefficient of all incident species. Furthermore, it carries a considerable uncertainty as it is based on a rough estimate of the flux of erosion products toward the slit of

the cavity. More detailed information results from the distribution of retained carbon atoms within the cavity.

For the case of physical sputtering Fig. 3(a) and (b) shows the distributions of carbon inside the cavity. A sharp peak of carbon is found on the bottom wafer beneath the entrance slit with C levels rapidly falling off to background levels, about 1×10^{15} cm^{-2}, to both sides.
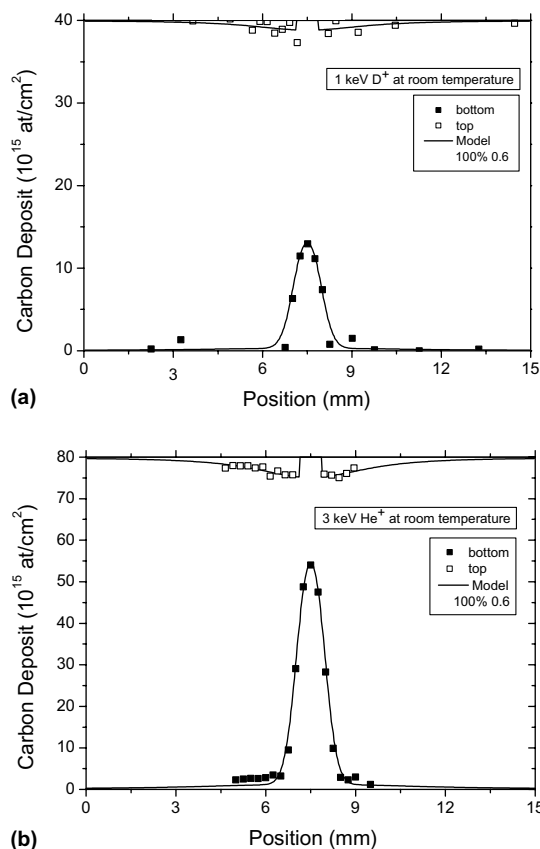

Fig. 3. Deposition pattern inside the cavity for conditions of physical sputtering: (a) 1 keV D$^+$ at room temperature and (b) 3 keV He$^+$ at room temperature.
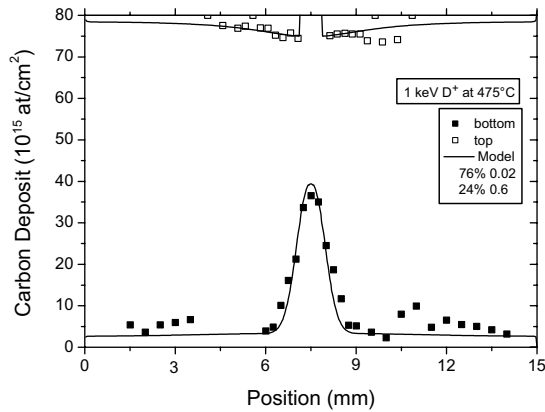
Fig. 4. Deposition pattern inside the cavity for conditions of predominantly chemical erosion, i.e. 1 keV D^+ at 475 °C.

However, on the inner side of the cover wafers, forming the entrance slit, clear carbon deposits were detected in the near vicinity of the slit, indicating a surface loss probability of the incident carbon atoms of less than unity.

In contrast to the conditions of physical sputtering the deposition pattern at 1 keV D^+ and 475 °C (Fig. 4) shows, in addition to the sharp peak, an almost uniform deposition in the wings of the distribution. To achieve agreement within the scatter of the data, a uniform carbon coverage of at least 2.5×10^{15} cm^{-2} has to be reproduced by the model. Therefore, at least two different eroded species with different surface loss probability on the collector surface have to be assumed.

The final condition investigated is chemical sputtering as described above. In this case, for 45 eV D^+ at room temperature, only a weak maximum beneath the entrance slit can be found, while the inner surfaces of the cavity are uniformly covered with 7×10^{15} cm^{-2} (Fig. 5).
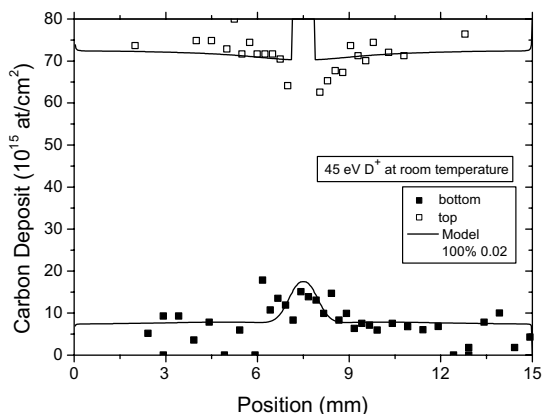

Fig. 5. Deposition pattern inside the cavity for conditions of chemical sputtering, i.e. 45 eV D^+ at room temperature.

This coverage is similar to the coverage at elevated temperatures, although the total amount of eroded carbon atoms was much less due to the smaller beam current at the low ion energies.

4.3. Modelling and evaluation

The surface loss probability of those species, which lead to deposition inside the cavity, can be obtained from a fit to the measured deposition pattern. The surface loss probability is defined as the total probability that the particle is not reflected from the wall as the original reactive species. That includes processes such as sticking and the transformation of radicals to saturated hydrocarbons.

Such fits are shown in Figs. 3–5 as solid line. The assumptions are a very narrow angular distribution of the particle beam entering the cavity, the sticking to the surface with a probability proportional to a pre-selected surface loss probability upon each collision with the surface and a cosine angular emission distribution of the reflected fraction. In cases with a uniform distribution on the cavity walls in addition to a sharp deposition peak beneath the slit contributions from deposited species with different surface loss probability have to be assumed. From the previous, quantitative comparison of the total amounts a lower limit of the sticking coefficient and thus the surface loss probability was derived to about 0.02. Therefore, all distributions were fitted with a combination of high-β species (0.6) and low-β species (0.02). The resulting fractions and surface loss probabilities are given in Table 2.

Both cases of physical sputtering, i.e. 1 keV D^+ and 3 keV He^+ at room temperature lead to the same result. Only one species has to be assumed having a surface loss probability of 0.6 to adequately describe the deposited distributions inside the cavity (Fig. 3(a) and (b)). This is surprising, as it can reasonably be assumed that sputtered carbon atoms have energies of several eV, resulting in a sticking coefficient close to unity. However, especially the amount of carbon found on the inner side of the entrance slits requires to allow for reflected particles. As the reflection coefficient of C from Si at these energies is much less than 0.4 [17], the re-erosion due to the simultaneously incident reflected sputtering ions must be assumed for this result. Estimates of this effect using the sputtering code TRIM.SP [17] just considering physical sputtering by reflected He ions yield about 16% re-erosion of the deposited carbon layer and do not quantitatively explain an effective sticking coefficient of 0.6. For D ions the additional effect of chemical sputtering at low ion energies might play a role.

In the case of chemical sputtering at low ion energies the distribution (Fig. 5) can be adequately described with two different assumptions: just one eroded molecular species having a surface loss probability around

Table 2
Composition of the eroded particle flux entering the cavity

	Physical sputtering 3 keV He⁺, RT; 1 keV D⁺, RT	Thermal chemical erosion 1 keV D⁺, 475 °C	Chemical sputtering 45 eV D⁺, RT
Surface loss prob. 0.6	100%	24%	0
Surface loss prob. 0.02	0	76%	100%
Sticking coefficient ≈ 0	0	Factor > 1.5 from weight loss	0

0.02 or two eroded species, one having again a surface loss probability of 0.6, but a majority of particles (71%) having a much smaller surface loss probability of the order of 0.001 or smaller. The second assumption can be ruled out on quantitative arguments: species with a surface loss probability lower than 0.001 have a high probability ($\geqslant 96\%$) to escape through the slits without being deposited within the cavity [12]. The detected fraction of low-β species has to be multiplied by a factor of $\geqslant 25$ (see Fig. 1), taking the escape probability into account and therefore, would by far exceed the total number of molecules entering the cavity, as estimated in Table 1. On the other hand, assuming one species with $s = \beta = 0.02$ and an escape probability of $\approx 1/2$ reproduces both, the distribution inside the cavity (Fig. 5) and the total number of carbon atoms within the cavity (Table 1). Such a sticking probability can be attributed to radicals with sp³ hybridisation, such as CD_3, on surfaces activated due to simultaneous incidence of atomic hydrogen [18,19], while radicals of hydrocarbons with sp²- or sp¹-hybridisation typically have surface loss probabilities around 0.35 or 0.8, respectively [13]. If we take into account the indirect flux of low-β species, the sticking coefficient could even be by about a factor of two lower. Alternatively, further species with negligible sticking, i.e. saturated hydrocarbons, might contribute with a flux comparable to that of the $\beta = 0.02$ species.

In the case of thermal chemical erosion combined with physical sputtering at 1 keV D⁺ and 475 °C surface temperature (Fig. 4) the amount of carbon atoms found inside the cavity is even much lower than the direct incident flux. This already indicates a low sticking probability of parts of the eroded species. The fraction of 24% of the species having a sticking coefficient close to 0.6 agrees well with the kinetically sputtered atom fraction in comparison to room temperature (Table 1). In addition, a second fraction of emitted species has a sticking coefficient equal or lower than 0.02, a value previously determined for CD_3 radicals [18,19]. Taking the escape probability of this fraction into account, their total contribution amounts to 76% of all deposited species in the cavity. However, from the relation of the number of atoms directly entering the cavity, estimated from the weight loss of the sample, to the number of entering atoms evaluated from Eq. (1) there remains a fraction of 50% which has to be attributed to volatile saturated hydrocarbon molecules with negligible stick-

ing on walls or to the transformation of reactive hydrocarbons to saturated ones upon wall contact, or in other words $s < \beta$. In both cases they could be readily observed in residual gas analysers. Actually, this fraction is rather low compared to literature values of about 80% saturated hydrocarbons and 20% CD_3 radicals measured using line-of-sight detectors for 2.5 keV D⁺ at 800 K [20]. However, while the ratio between the contributions of the species with $\beta = 0.6$ and 0.02 is rather precisely determined by the deposition profile, the contribution of species with negligible sticking depends on an estimation of the carbon influx into the cavity. The dominant error is clearly the indirect flux of low-β species into the cavity.

The present results are in good agreement with previous investigations comparing open collector probes, weight-loss measurements and residual gas analysis (RGA) of produced species [16]. It was previously shown that in the case of low energy, low temperature chemical sputtering about 50% of the sputtered species could be collected on material probes around the target while residual gas analysis was able to detect only a fraction of the total erosion evaluated from the measured weight loss. The present investigation shows considerable contributions of radicals with a surface loss probability around 0.02. Such radicals will have good chances to be lost to vessel walls before reaching an RGA, which is not in line of sight to the target.

High-temperature chemical erosion results in comparable emissions of low-β radicals (0.02), and volatile saturated hydrocarbons, which will not be readily deposited on the vessel walls, but can be pumped away and be detected efficiently in RGAs. Taking into account that low-β or zero-β radicals enter the cavity also indirectly, the relative contribution of volatile saturated hydrocarbons must dominate under these conditions of erosion in agreement with information on the contributions of CD_4 and heavier saturated hydrocarbons, available from RGA not in line of sight to the target and corresponding to weight-loss measurement [16].

As a consequence of the present investigations, it becomes clear that ion energy and surface temperature are the dominant parameters determining not only the erosion yield, but also the re-deposition of carbon layers in fusion devices. As can be seen from the retention inside the cavity given in Table 1 low energy, low temperature erosion appears in this respect to be most

efficient in carbon layer and tritium inventory build-up. The influence of other important parameters, such as the surface temperature of the collector surface, will be the topic of further investigations.

5. Conclusion

The combination of quantitative erosion measurements in mass-analysed ion beams and the use of cavities as collectors for the eroded species enables the determination of surface loss probabilities of the eroded species and an estimation of a lower limit for the sticking coefficient.

Different sputter erosion processes of carbon lead to different eroded species with widely differing surface loss probabilities and sticking coefficients. The cavity technique has resulted in the following findings:

- While physical sputtering produces carbon atoms with surface sticking coefficients close to unity, the situation is different for hydrogen ion bombardment; in this case hydrocarbon radical emission has to be taken into account.
- During chemical sputtering at room temperature and low ion energies radicals are being emitted which have low surface loss probabilities around 0.02. Such values are typical of sp^3 hybridized radicals, possibly CH_3, on activated surfaces [13,18,19].
- Thermal chemical erosion at elevated temperatures leads to comparable amounts of emitted species with a sticking coefficient around 0.02 and 0, tentatively being attributed to CH_3 radicals and saturated hydrocarbons, respectively.

In fusion devices the chemical sputtering process, i.e. the erosion at ion energies below 50 eV and surface temperatures below 100 °C will lead to the emission of radicals with sticking coefficients on Si-collector surfaces of the order of 0.02. These radicals can survive several tens of surface collision before being permanently trapped. Hydrocarbons with surface loss probabilities of the order of 10^{-3}–10^{-4} could not be found. They are judged most dangerous for the build-up of carbon layers in poorly accessible, remote areas retaining T by co-depo-sition. However, s and β also depend on the surface condition and can be significantly smaller on less activated surfaces. The remaining emitted species are saturated hydrocarbons which can be efficiently pumped.

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