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Chemical erosion of carbon based materials in fusion devices

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Abstract

In the last two years the basic erosion mechanisms of carbon due to thermal and energetic hydrogen atoms have been resolved and the range of erosion yield data has been extended into new regimes of ion flux and energy. The present paper reviews the recent achievements in fundamental understanding. Model prediction of the dependence on ion energy and flux are compared with new experimental results from fusion experiments and plasma simulators. For graphites doped with B or Si the decrease of the erosion yield as function of energy and temperature indicates which elemental steps in the erosion process are influenced. © 1999 Elsevier Science B.V. All rights reserved.

Keywords: Carbon; Composite materials; Chemical erosion; Flux dependence; Ion irradiation

1. Introduction

Carbon plasma facing components such as divertor plates or protection limiters appear to be unequalled by any other material in future fusion devices due to their superior thermo-mechanical properties [1]. However, erosion due to low energy ion impact is high for all low-*Z* materials and, especially for carbon, the chemical reactivity with hydrogen atoms further enhances the erosion. This limits the lifetime of the components, dilutes the plasma with impurity ions and leads to high tritium inventories in deposited layers on less exposed areas of the vessel wall.

Chemical erosion has been the subject of many investigations under well-controlled conditions using ion beams in the energy range of 10 eV to keV and fluxes between 10^{18} and 10^{20} m^{-2} s^{-1} as reviewed at the 12th PSI Conference [2]. The conditions for plasma facing components require, however, the extrapolation to fluxes of the order of 10^{23}–10^{24} m^{-2} s^{-1} and energies even smaller than achievable in ion beam experiments. This extrapolation over many orders of magnitude can only be achieved with a good understanding of the basic mechanisms leading to the hydrocarbon release under hydrogen bombardment.

Since the last reviews on chemical erosion of carbon in 1996 [2,3] considerable improvements have been reached in the fundamental understanding. The thorough investigations by Horn et al. [4] on the thermal chemical reactivity has been extended to the case of energetic ion bombardment by Roth and García-Rosales [5]. Simultaneously, new ion beam experiments at energies down to 10 eV [6–8] have improved the understanding of ion induced hydrocarbon emission. Very recently, dedicated spectroscopical investigations on the chemical erosion in the ASDEX Upgrade divertor [9,10] as well as controlled erosion studies in the Berlin Plasma Generator PSI-1 [11,12] have provided data in an energy and flux regime close to the ITER divertor conditions.

The present paper will review the physical understanding of chemical erosion and compare the analytic modeling [5] to the available data base. The trends and absolute values of the new low ion energy and high flux data will be discussed and used to improve the analytic model.

2. Thermal and ion induced mechanisms of chemical erosion

Three processes determine the chemical erosion of carbon under low energy hydrogen bombardment:

(1) The *reaction of thermalised ions* within the implanted surface proceeds via the hydrogenation of car-

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bon atoms to CH_3–C complexes. At temperatures above 400 K CH_3 radicals can be released while at temperatures above 600 K recombinative hydrogen release (H_2) starts to reduce the chemical erosion yield. This thermal chemical erosion was elucidated in detail by Küppers et al. [4,13] and was described analytically by Roth and García-Rosales [5]. For the thermal reaction no dependence on the hydrogen isotope was observed.

(2) The thermal reaction is *enhanced by radiation damage* introduced in the material which provides open bonds for hydrogen attachment. Damage is created by kinetic energy transfer from incident ions to lattice atoms and is, therefore, responsible for the dependence of the chemical erosion yield on hydrogen isotope. This yield enhancement is characterized by a threshold energy for damage production, E_{dam}. The basic thermal reaction below the threshold for damage production depends strongly on the crystalline perfection of the carbon material with maximum yields between 10^{-3} for well annealed pyrolytic graphite and 10^{-1} for amorphous a-C:D layers [14]. At energies where radiation damage amorphises the graphite lattice, the strong dependence on the material structure disappears [15].

(3) At low surface temperatures all available carbon atoms are essentially hydrated but no thermal release of hydrocarbons occurs. However, hydrocarbon radicals are bound to the surface with much smaller binding energy (≈1 eV) than carbon surface atoms in their regular lattice environment (7.4 eV). This leads to an *ion induced desorption of hydrocarbon radicals* which can be described in a manner analogous to physical sputtering using a threshold energy, E_{des}, in the low eV range.

The investigation and description of these processes has been performed for thermal hydrogen atom or ion fluxes of the order of 10^{18}–10^{20} m^{-2} s^{-1}. The thermal reaction cycle predicts a pronounced shift of the temperature, T_{max}, where the maximum yield occurs towards higher temperatures with increasing ion flux, while the maximum yield itself decreases only slightly. This temperature shift with ion flux is well reproduced in the experimental data [16]. However at fluxes above 10^{21} m^{-2} s^{-1} as reached in plasma simulators or under tokamak conditions, the temperature of maximum yield does not exceed 900 K. At these elevated temperatures, the thermodynamic equilibrium of H/C system shifts from CH_4-formation to H_2 release [17] and the erosion yield is expected to decrease with ion flux. Additionally, annealing of radiation damage will result in lower reactivity of the carbon material.

3. Quantitative comparison with experimental results

The above model has been formulated analytically by Roth and García-Rosales [5]. However, in the light of new data at low ion energies several parameters of the ion induced processes can be more quantitatively obtained. The results by Mech et al. [6,18] confirmed the validity of the assumed erosion processes and allowed the quantitative determination of the threshold energies for desorption, E_{des}, and for damage production, E_{dam}. Fig. 1 shows the comparison of the determined cross sections for ion induced desorption and damage production obtained by Mech et al. [18] with the analytic description of these processes analogous to physical sputtering. Clearly, E_{dam} is much lower than 30 eV and E_{des} seems to be almost independent of the isotope. Values of 2 eV for E_{des} and 15 eV for E_{dam} both for H

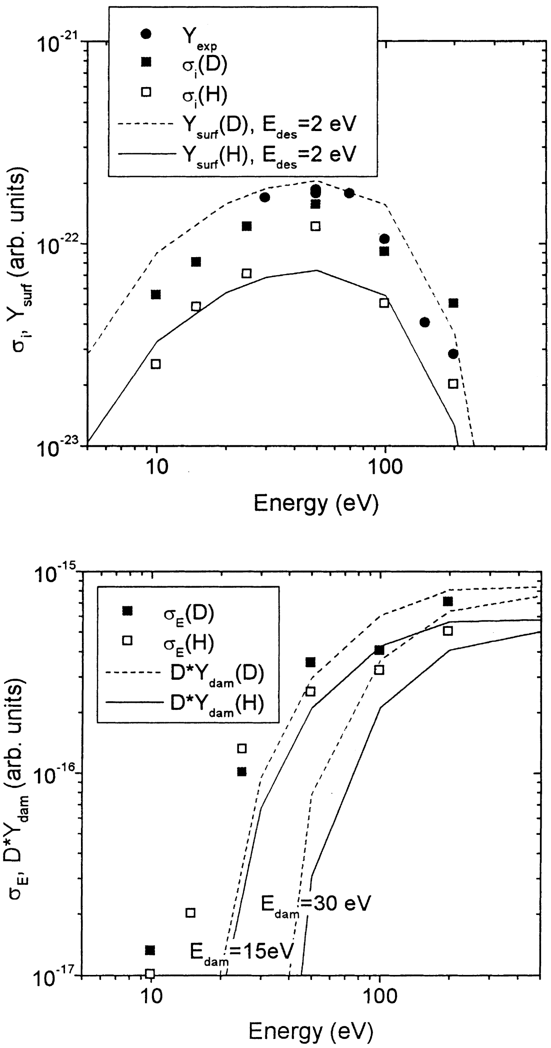

Fig. 1. (a) Comparison of experimentally determined cross sections for sputtering of surface radicals [7] with the analytic function for a threshold energy, $E_{des} = 2$ eV. (b) Comparison of experimentally determined cross sections for radiation damage [7] with the analytic function for two different threshold energies, E_{dam}.

and D ions are deduced from the data by Mech et al. [18] and the data collection from previous low energy results [5].

The kinetic nature of the emission of radicals at low ion energies has been demonstrated by collector experiments by Roth and Balden [8]. They showed that only 50% of the emitted species can be detected as saturated CH_4 molecules in residual gas analysis and about 50% of the eroded carbon atoms were retained on Al-collector strips in front of the target. For grazing angle of incidence the angular distribution of emitted hydrocarbon radicals shows clearly the anisotropic forward distribution as found for physical sputtering. The fact that emitted hydrocarbon radicals partly stick to the vessel surfaces rather than being hydrated to volatile saturated hydrocarbons may explain the experimental discrepancies between erosion yields determined by weight loss and by residual gas mass spectrometry (see Fig. 2).

Another improvement in the analytic description was stimulated by the detailed measurements of the temperature dependence at low ion energies [6,18]. As already suggested by Horn et al. [4] this temperature dependence cannot be adequately described by single activation energies but rather by distributions of activation energies with a standard deviation of 0.3–0.5 eV. The single activation energies assumed for simplicity by Roth and García-Rosales [5] lead to abrupt transitions from the temperature-independent processes to the thermal chemical yields which are not supported by the experimental data.

With these improvements, a modified set of equations [19] can be given describing the total erosion yield of carbon. The detailed equations for each erosion process and a table of the parameters involved are given in Appendix A. In Fig. 2 the analytic model is compared to a collection of yield data [5,6,20–25] as function of temperature for 50 and 200 eV ion energy and fluxes of 1×10^{19} and 1.5×10^{22}, respectively. The experimental data show large scatter, in general within a factor of 2, due to different material structure of the graphite and different measuring methods, with weight loss measurements always resulting in higher yields than mass spectrometric data. However, the general trends with ion energy, temperature and flux are well-described by the modeling results. The broadening of the temperature dependence with ion flux, the shift of T_{max} to higher temperatures and the decrease of the yield at T_{max} for high fluxes are especially well reproduced.

4. Interpretation of new experimental data

For the extrapolation to higher fluxes, very recently, two sets of experimental data became available which will be presented in detail at this conference:

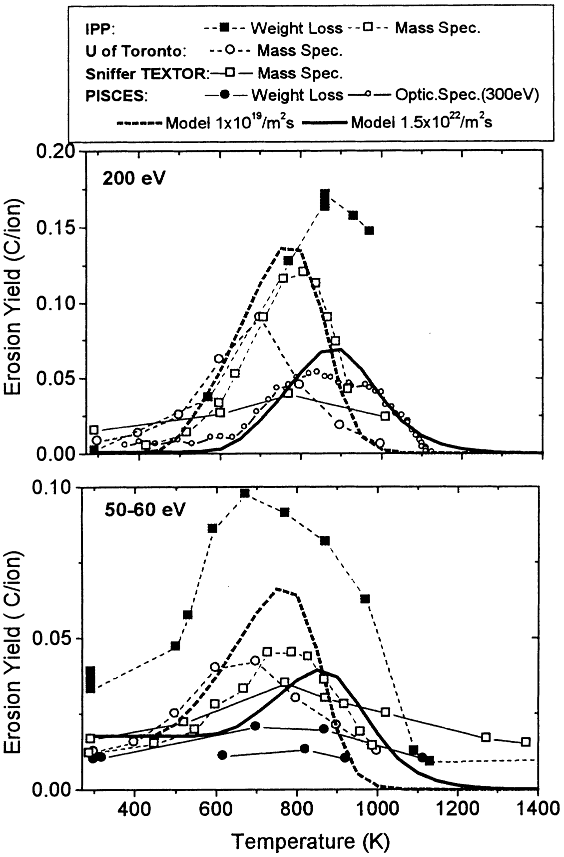

Fig. 2. Comparison of experimental data for the temperature dependence of the chemical erosion of carbon under deuterium bombardment with the analytic model outlined in Appendix A. Data for ion fluxes around 10^{19} m^{-2} s^{-1} (ion beam experiments): dashed lines; high flux data around 1.5×10^{22} m^{-2} s^{-1} from plasma experiments: solid lines. Note that physical sputtering has been subtracted from weight loss data.

(1) In the divertor of ASDEX Upgrade the chemical erosion yield was determined spectroscopically for a variety of plasma conditions [9,10]. The ion flux and the emitted hydrocarbons were quantitatively determined from the H_β and H_γ intensity as well as from the CH and CD band intensity, respectively. As the surface temperature remained constant between 300 and 400 K and the incident ion energy was between 10 and 100 eV, the flux and energy dependence of Y_{surf} were investigated.

(2) Furthermore, at the plasma generator PSI-1 in Berlin the thermal chemical erosion yield at the temperature maximum, T_{max}, was investigated as a function of ion flux at constant plasma temperature between 4 and 6 eV, corresponding to ions accelerated in the sheath potential to energies between 20 and 30 eV [11]. For the yield evaluation three different methods, i.e. CD spectroscopy, mass spectrometry and weight loss, were

used. Again, values obtained by weight loss in general exceed spectroscopic data; however, within the experimental errors the values agree well. The calibration of optical emission spectroscopy and mass spectrometry was done by calibrated methane puffing to avoid uncertainties due to the strong dependence of dissociation, excitation and ionization processes on plasma conditions [12].

Both sets of data show a pronounced dependence on ion flux. However, in the case of the ASDEX Upgrade data, variations of the flux correspond always to variations in the incident ion energy, such that an increase in flux is correlated to a decrease in ion energy. The average incident particle energy was determined from the ion flux and thermographic observations of the power deposition to the surface as well as from Langmuir probes embedded in the divertor plates [10]. Within the observed range of ion energies of 10–100 eV, a considerable energy dependence of the surface process, Y_{surf}, is predicted (see Fig. 1(a)). A comparison of the experimental data with model calculations as function of energy shows that the dependence of the erosion yield on the hydrogen isotope is well reproduced while the energy dependence is obscured in the scatter of the data. The large scatter of the data may partly be due to uncertainties in the evaluation of the average ion energy or to changing plasma parameters, such as during ELMs in H-mode discharges. However, if plotted against the ion flux (Fig. 3(b)), this scatter of the data is greatly reduced and a decrease of the yield with flux according to $\Phi^{-0.7}$ is obtained. This good correlation suggests a strong additional dependence on ion flux besides the additional dependence on ion energy [10].

In Fig. 3(a) the results from PSI-1 at $T_e \approx 5$ eV and previous data from TEXTOR [26] at $T_e \approx 40$ eV at the maximum temperature for chem erosion, $T_{\max}$, are compared to the model predictions as a function of ion flux. Although obscured by large error bars and a large scatter due to different discharge conditions the data confirm the general trend of a decrease of the maximum yield with ion flux as predicted by the model. Actually the slow decrease with flux in the range between 10^{18} and 10^{21} m^{-2} s^{-1} is accelerated for fluxes above 10^{21} m^{-2} s^{-1}. The new data from the PS 1 indicate clearly an even steeper decrease with ion flux than predicted, similar to the room temperature data from ASDEX Upgrade.

The model was derived on the basis of steady state balance equations for the different carbon configurations and did not take into account reaction times which in polymers are known to be exceedingly long. In conjunction with the data from ASDEX Upgrade it can be speculated that an additional flux dependence may be a consequence of time consuming rearrangements of chemical bonds in the hydrogenation process of carbon atoms to hydrocarbon radicals as the precursor for both, the high temperature chemical erosion and for low temperature sputtering of radicals. A time constant has the consequences that at high fluxes the surface population of CH_3–C complexes is limited by the hydrogenation process such that the emitted particle flux becomes independent on ion flux and the erosion yield decreases with $1/\Phi$. With the assumption that the effective cross section for erosion, σ_y, is always small compared to the hydrogenation cross section, this leads to a correction term of the order of $1/(1 + \sigma_y \Delta t \Phi)$. The best fit of this function to the measured flux dependence of Kallenbach et al. [10] and Grote et al. [11] results in $\sigma_y \Delta t \approx 3 \times 10^{-23}$ m^2 s. The erosion cross section σ_y can be estimated from the ion range and the erosion yield to about 10^{-22} m^2 such that the hydrogenation time is of the order of 1 ms. Similarly long time constants are actually deduced from the dependence of hydrogenation on the deposition rate during the growth of a-C:H layers [27]. The proposed correction term taking the effect of a long hydrogenation time into account is given by Eq. (A.7) in Appendix A.

In the light of the prevailing uncertainties of the new experiments and awaiting the extension of the PSI-1 data to higher fluxes and lower surface temperatures, the yields recommended by the ITER expert group [28] for estimating erosion in high flux divertor application [29] take a more conservative approach. In the yield range between 10^{21} and 10^{23} m^{-2} s^{-1} no flux dependence was assumed. In Fig. 3 the available data are compared to the different model predictions and estimates. The procedure for arriving at the recommendations is described in detail in Ref. [30].

5. Influence of doping on different chemical erosion mechanisms

The complex thermal chemical reactivity cycle is strongly dependent on the electronic structure of carbon in the graphite lattice. It is known that this electronic structure can be sensitively influenced by additions of substitutional boron [31]. Consequently, the thermal chemical erosion could be strongly reduced by small additions (<3 at.%) of boron to the graphite [20] and also additions of Si, Ti and W have shown reductions of the yield on the order of a factor of 2 [32–34]. On the other hand, the surface erosion process, Y_{surf}, for boron doped graphites could not be reduced by the same amount, indicating that it is not the hydrogenation of carbon atoms at low temperatures which is influenced. The temperature dependence of the chemical erosion shows no influence on boron doping up to temperatures of about 700 K while at higher temperatures the erosion is almost suppressed. This, together with the finding that the thermal desorption of H_2 from implanted boron doped graphite occurs at temperatures about 150 K lower than for undoped graphite [35] shows clearly, that

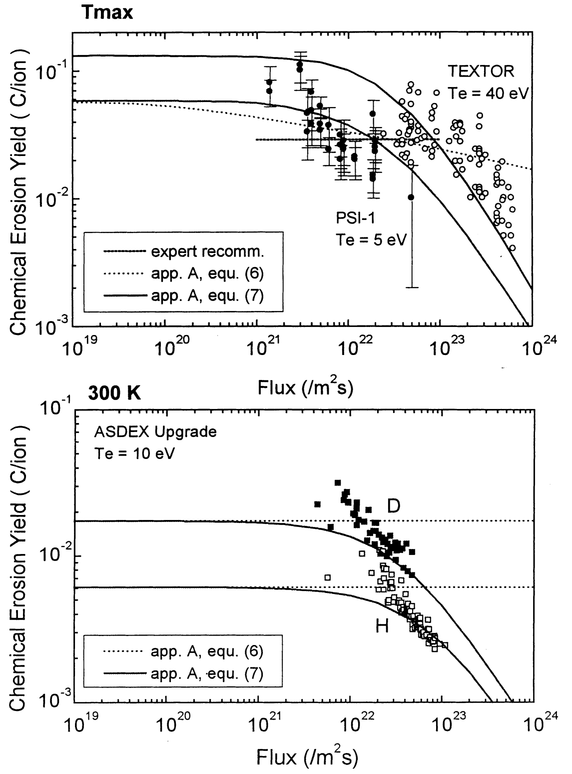

Fig. 3. Dependence of the chemical erosion yield on ion flux for surface temperatures around T_{max} (top) and room temperature (bottom). Experimental data are compared with predictions from the analytic model outlined in Appendix A. For comparison, the expert recommendations [28] for an ion energy of 20 eV at T_{max} are also given.

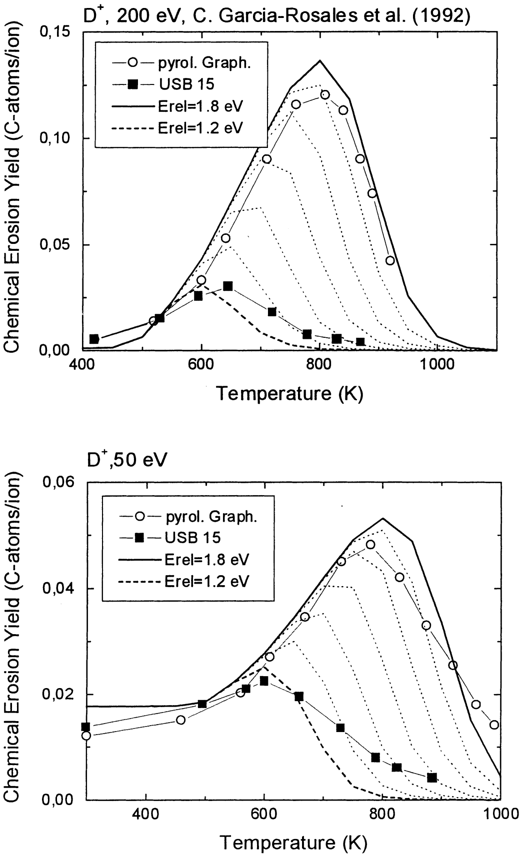

Fig. 4. Temperature dependence of the hydrocarbon yield for pure carbon and USB 15, a graphite with 15 at.% B [20]. The data are compared with predictions from the analytic model outlined in Appendix A for a variation of the activation energy for hydrogen release, E_{rel}, in steps of 0.1 eV.

the doping enhances the recombinative release of hydrogen in the form of H_2 molecules. Therefore, the analytic model for chemical erosion has been applied to the case of doped graphites just by reducing the activation energy for hydrogen release, E_{rel}, which is about 1.8 eV for pure graphite. In Fig. 4 the temperature dependencies for chemical erosion for pure graphite, USB15 (15 at.% B) for the case of 50 and 200 eV D bombardment is compared with model calculations for various activation energies for hydrogen release. It can be deduced that for B doping the activation energy decreases to 1.2 eV with all other parameters unchanged.

Different dopants may, however, influence the erosion cycle in different ways. In the case of Si, no similar shift in the temperature of the hydrogen desorption could be observed with respect to pure carbon [35]. Consequently, a much smaller reduction of the chemical erosion is observed for doping with Si, Ti, and W [32–34]. A fit to the erosion data by Chen et al. [34] results in a much smaller reduction of the activation energy for hydrogen release, E_{rel}. The best fit for Si, Ti, and W doped graphites is $E_{rel} = 1.5$ eV. Very importantly the distribution of dopants in the lattice is a decisive quality, which is strongly dependent on the doping procedure, the microstructure of the bulk material and on the final temperature treatment. In general, a very homogeneous atomic or fine grain doping will show the best results [33]. Dopants in the form of larger precipitates will show minor yield reductions, comparable to the reduction observed in the surface concentration of carbon.

At low energies and low temperatures no pronounced reduction of hydrocarbon emission due to the doping with Si or B is observed. However, as carbon is eroded preferentially, an enrichment of the dopants must be expected within the surface layer, ultimately leading to a reduction of the carbon erosion at high ion fluences. The higher the threshold energy for physical sputtering of the dopant atoms, the more pronounced this effect is. Consequently, dopants with higher atomic mass such as Ti will prove more advantageous and only for Ti is a strong reduction of the surface erosion term observed [33]. This enrichment of the dopant will, again, be reached faster for very fine grain structure or atomically homogeneous distribution of atoms.

6. Conclusions

The basic atomic processes of chemical erosion of graphite due to energetic hydrogen ions at ion fluxes below 10^{22} m^{-2} s^{-1} are well understood and analytic modeling adequately describes the experimental data base. At fluxes larger than 10^{22} m^{-2} s^{-1} the model describes well the general trend of a shift of $T_{\max}$ to higher temperatures and a decrease of the maximum yield. Under divertor conditions, i.e. at high fluxes and low ion energies erosion yields will be of the order of 1% or below. New experimental results in this flux range, suggesting a steeper decrease of the maximum yield with ion flux, may require new modeling efforts. The current data base is still unsatisfactory, but suggests the existence of a flux dependence due to a finite time constant in the hydrogenation of carbon atoms.

The extrapolation of the thermal chemical erosion process to particle energies below 5 eV down to thermal atoms depends strongly on the microstructure of the carbon material and on damage due to energetic ions. Physical sputtering does not occur in this ion range and thermal yields range between 10^{-3} and 10^{-1} for low fluxes. Erosion yields for redeposited layers tend to be on the high side of the yield range. The erosion yield for very low energy ions or thermal hydrogen atoms may depend on the location in the divertor, i.e. whether the surface is erosion or redeposition dominated.

Doping can strongly reduce the thermal chemical reactivity, while ion induced surface desorption of hydrocarbons can only be reduced by dopant enrichment at high fluences. Atomic or fine grain doping, especially by high-Z atoms, may prove most efficient but must be limited to low concentration to prevent plasma contamination.

Appendix A. Analytic equations for erosion of carbon under hydrogen bombardment (J. Roth, H. Pacher, NET Team, Garching)

The total erosion yield of carbon under hydrogen bombardment, Y_{tot}, is composed of three components [5]: the physical sputtering yield, Y_{phys}, the thermal erosion yield, Y_{therm}, enhanced by radiation damage and the sputtering of radicals, Y_{surf}:

$$Y_{\mathrm{tot}} = Y_{\mathrm{phys}} + Y_{\mathrm{therm}}(1 + DY_{\mathrm{dam}}) + Y_{\mathrm{surf}}, \tag{A.1}$$

where D is a parameter depending on the hydrogen isotope.

The physical sputtering yield for an ion energy E_0 is given by

$$Y_{\mathrm{phys}}(E_0) = QS_n(E_0)\left[1 - \left(\frac{E_{\mathrm{th}}}{E_0}\right)^{2/3}\right]\left(1 - \frac{E_{\mathrm{th}}}{E_0}\right)^2 \tag{A.2}$$

with

$$S_n(E_0) = \frac{0.5\ln\left[1 + 1.2288(E_0/E_{\mathrm{TF}})\right]}{E_0/E_{\mathrm{TF}} + 0.1728\sqrt{E_0/E_{\mathrm{TF}}} + 0.008(E_0/E_{\mathrm{TF}})^{0.1504}}. \tag{A.3}$$

The thermal erosion yield at an ion flux, Φ, can be obtained by

$$Y_{\mathrm{therm}} = c^{\mathrm{sp}^3}\frac{0.033\exp\left(\frac{-E_{\mathrm{therm}}}{kT}\right)}{2\times10^{-32}\Phi + \exp\left(\frac{-E_{\mathrm{therm}}}{kT}\right)} \tag{A.4}$$

with

$$c^{\mathrm{sp}^3} = \frac{C\left[2\times10^{-32}\Phi + \exp\left(\frac{-E_{\mathrm{therm}}}{kT}\right)\right]}{2\times10^{-32}\phi + \left[1 + \frac{2\times10^{29}}{\Phi}\exp\left(\frac{-E_{\mathrm{rel}}}{kT}\right)\right]\exp\left(\frac{-E_{\mathrm{therm}}}{kT}\right)} \tag{A.5}$$

and

$$C = \frac{1}{1 + 3\times10^{7}\exp\left(\frac{-1.4}{kT}\right)}, \tag{A.6}$$

where the flux, Φ, is given in ions/m^2 s. If for high ion fluxes a possible influence of the hydrogenation time is taken into account, the correction term C could be replaced by

$$C = \frac{1}{1 + 3\times10^{-23}\Phi}. \tag{A.7}$$

Future data at higher fluxes will allow us to determine whether a combination of Eqs. (A.6) and (A.7) will be more adequate.

The thermal chemical erosion is enhanced by damage production given by

$$Y_{\mathrm{dam}}(E_0) = QS_n(E_0)\left[1 - \left(\frac{E_{\mathrm{dam}}}{E_0}\right)^{2/3}\right]\left(1 - \frac{E_{\mathrm{dam}}}{E_0}\right)^2. \tag{A.8}$$

The surface erosion process is quantified by

$$Y_{\mathrm{surf}}(E_0, T) = c^{\mathrm{sp}^3}\frac{Y_{\mathrm{des}}(E_0)}{\left[1 + \exp\left(\frac{E_0 - 65\ \mathrm{eV}}{40}\right)\right]}, \tag{A.9}$$

where

$$Y_{\mathrm{des}}(E_0) = QS_n(E_0)\left[1 - \left(\frac{E_{\mathrm{des}}}{E_0}\right)^{2/3}\right]\left(1 - \frac{E_{\mathrm{des}}}{E_0}\right)^2. \tag{A.10}$$

Table 1
Parameters for the chemical erosion by different hydrogen isotopes

Parameter	Hydrogen	Deuterium	Tritium
E_{TF}	415 eV	447 eV	479 eV
Q	0.035	0.1	0.12
E_{th}	31 eV	27 eV	29 eV
E_{dam}	15 eV	15 eV	15 eV
E_{des}	2 eV	2 eV	2 eV
E_{rel}	1.8 eV for pure carbon 1.5 eV for Si, Ti, W doped carbon 1.2 eV for *B* doped carbon		
E_{therm}	Gauss distribution of activation energies around 1.7 eV, $\sigma = 0.3$ eV		
D	250	125	83

The parameters Q, E_{therm}, E_{th}, E_{dam}, E_{des}, E_{rel}, E_{TF} and D are given in Table 1 for the different hydrogen isotopes. Changes in the numerical values in Eqs. (A.6) and (A.9) compared to Ref. [5] are only introduced to smoothen the transition between different processes.

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