Charge exchange, surface-induced dissociation and reactions of doubly charged molecular ions SF_4^{2+} upon impact on a stainless steel surface: A comparison with surface-induced dissociation of singly charged SF_4^+ molecular ions[☆]

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ABSTRACT

Collisions of SF_4^{2+} and SF_4^+ ions with hydrocarbon-covered stainless steel surface at room temperature were investigated. The projectile ions were mass selected by a two-sector-field mass spectrometer and decelerated to incident energies of 60 to a few eV. Product ions were measured with the use of a time-of-flight spectrometer and their relative abundances determined as a function of the incident energy of the projectile ions (collision-energy-resolved mass spectra, CERMS curves). The mass spectra of product ions were dominated by fragment ions SF_3^+, SF_2^+, and SF^+ at incident energies below 40 eV, while sputtering of contaminant adsorbates prevailed at higher energies. The results indicate that the likely major reaction sequence responsible for the observed CERMS curves of product ions from SF_4^{2+} collisions is charge exchange to form singly charged projectile ions followed by subsequent unimolecular fragmentation. In addition, chemical reactions between projectile ions and hydrocarbon adsorbates were observed leading to $SF_2CH_3^+$, $SFCH_2^+$, and SCH^+ ions.

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1. Introduction

Studies of ion–surface collisions are a research area which has undergone rapid growth in the past 20 years. Considerable interest has been devoted to studying selected physical and chemical processes stimulated by the impact of slow ions of incident energies up to 100 eV [1–5]. In this incident energy regime the energy transferred to the surface is of the same order of magnitude as energies observed in chemical bonds. Thus, slow ion–surface interaction studies can provide useful information regarding the nature of both, the projectile and the surface, as well as the characteristics of ion–surface interaction, i.e., surface-induced dissociation (SID), charge exchange reactions (CER) and surface-induced reactions (SIR). SID has been developed as an alternative to gas-phase collision-induced dissociation (CID) for the fragmentation of polyatomic ions in tandem mass spectrometry [3,4,6–8].

Besides being of fundamental importance, ion–surface collisions are also relevant to technological applications, such as plasma–wall interactions in electrical discharges and fusion plasmas [9]. Moreover, molecular photoionization has been suggested as a source of energetic charged particles in the terrestrial ionosphere and in the interstellar medium [10] and these charged particles are assumed to react on the surface of dust grains. In certain cases single photon absorption can lead to the ejection of two valence electrons [11] and thus to the production of doubly charged ions.

Charge transfer between the surface and the impacting projectile plays an important role in many processes involving the interactions of atoms and molecules with surfaces. The scattering event may be quite different for differently charged projectiles due to the dependence of the surface potential on the charge state of the projectile [1]. Past studies on reactive interactions have been dominated by the use of singly charged molecular ions and only two previous studies [12,13] on doubly charged molecular ion behaviour have been reported so far. That is unfortunate, not only for the intrinsic interest on such species [14], but also because in the case of singly charged ions, the charge transfer reaction leads to neutral products which are generally difficult to observe. Alternatively,
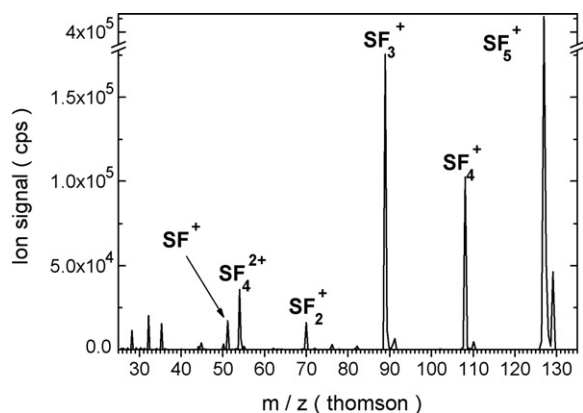
Fig. 1. Primary ion mass spectrum of sulphurhexafluoride (SF_6) ionized by 94 eV electrons.

with multiply charged ions, single charge exchange leads to charged product ions that can be easily identified and detected. Moreover, a comparison of the fragmentation pattern produced by singly and doubly charged molecular projectiles might give additional insight into the interaction of multiply charged ions with a surface.

Recently, we have started a series of measurements to investigate charge exchange, SID and SIR of small triatomic dications CO_2^{2+}, COS^{2+}, CS_2^{2+} and $CHCl^{2+}$ [15,16] with surfaces, followed by studies on SID and SIR of larger polyatomic dications $C_4H_3^{2+}$, $C_3H_5^{2+}$ [16], $C_6H_5^{2+}$, $C_6H_6^{2+}$ [12,17] $C_7H_8^{2+}$, $C_7H_7^{2+}$ and $C_7H_6^{2+}$ [13,18,19] investigated in the incident energy range from a few eV to 50 eV. Depending on the projectile ion distinct trends in the charge exchange and SID of these dications were found: e.g., (i) in the case of CO_2^{2+} and COS^{2+}, extensive fragmentation occurs with low collision energy thresholds as compared to the respective singly charged ions, probably due to charge separation reactions; (ii) double charge exchange with the surface, in the case of CS_2^{2+}, $CHCl^{2+}$, $C_3H_5^{2+}$, leads to complete neutralization; (iii) in the case of $C_4H_3^{2+}$,
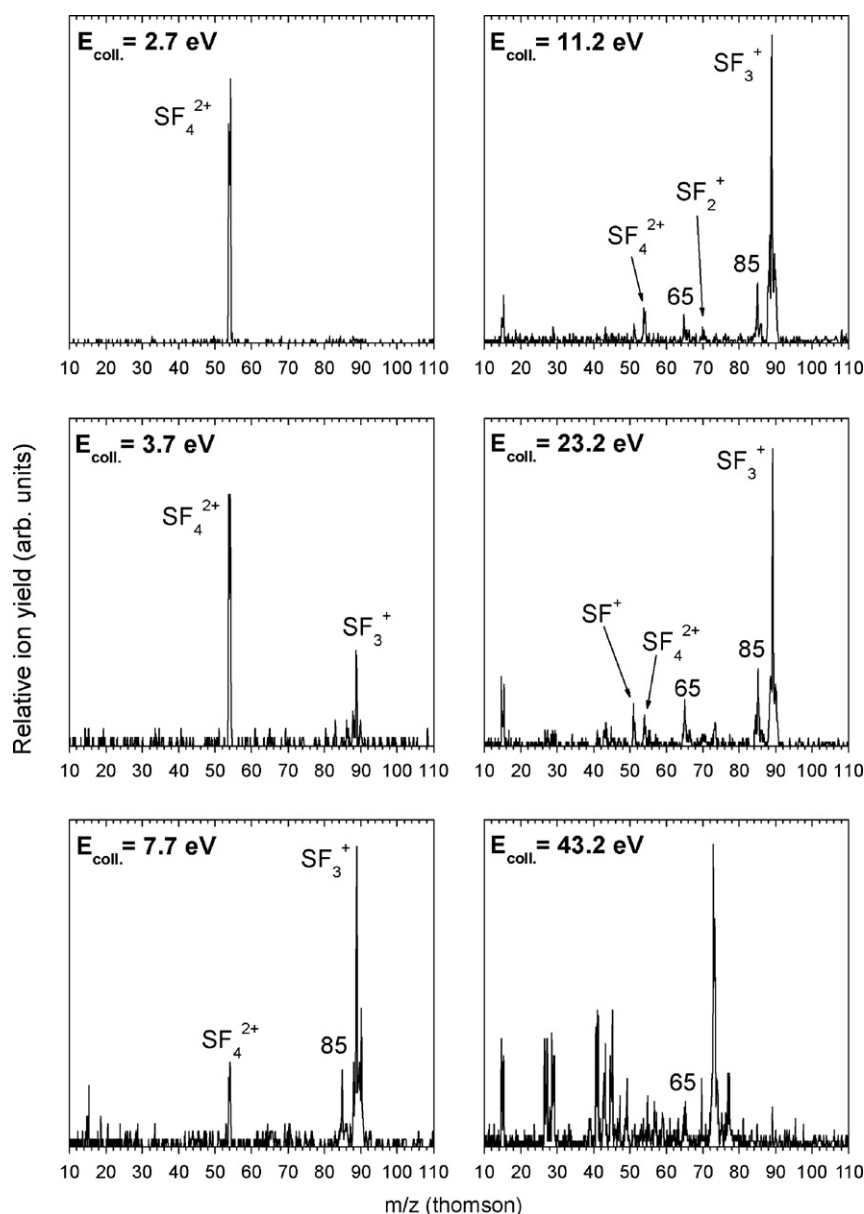

Fig. 2. Product ion mass spectra generated by the surface impact of doubly charged ions SF_4^{2+} at collision energies of 2.7, 3.7, 7.7, 11.2, 23.2 and 43.2 eV.

$C_6H_6^{2+}$, $C_6H_5^{2+}$, $C_7H_8^{2+}$, $C_7H_7^{2+}$ and $C_7H_6^{2+}$, single charge exchange between the dication and the surface can lead to unfragmented, singly charged parent ions, or be is followed by unimolecular decomposition reactions.

Sulphurhexafluoride (SF_6) is a man-made compound with many technological applications ranging from gaseous dielectrics to plasma etching [20,21]. It is also a very potent greenhouse gas, which is emitted during the etching process used in semiconductor industry [22,23]. A comparative study (1985–1994) of changes in the concentration of ozone depleting or potent greenhouse compound in the lower stratosphere (at altitudes between 17 and 30 km) showed an annual rate of increase for SF_6 of $8.0 \pm 0.7\%$ [24].

In the field of ion–surface collisions, projectiles consisting of fluorinated molecules are interesting because of the observed higher sputtering yield as compared to other isobaric species [25–28]. This effect has many practical applications and has been attributed on the one hand to purely kinematic mechanisms at high energies [26], or to charge-exchange reactions on the other hand [28].

Regarding SF_4^{2+}, Märk and coworkers [29,30], using the mass analyzed ion kinetic energy (MIKE) technique, measured the kinetic energy release for the Coulomb explosion of SF_4^{2+} (produced by electron impact ionization of SF_6) leading to the formation of SF_3^+ and F^+ fragments with an average kinetic-energy release of about 5 eV. The measured appearance energy of 43 eV for the SF_4^{2+} ions formed by electron impact ionization of SF_6 and 45.5 eV for the SF_3^+ ions resulting from the decay of the SF_4^{2+} ions indicated that the SF_4^{2+} ions from SF_6 were formed in more than one state and

only the SF_4^{2+} ions formed in the slightly higher-lying state underwent a Coulomb explosion. Thus, a stable state of this dication exists, too. Also, gas-phase collisions of many molecular dications, including SF_4^{2+}, have been studied by Price and co-workers [31,32], who identified interesting bond-forming reactions with the target species, as well as neutral loss leading to smaller dications. For the sake of our discussion, it is worth noting that some information is available on the thermochemistry of SF_x singly charged ions [33–35], and that the decay of SF_4^+ has been studied by photoionization mass spectrometry and photoion–photoelectron coincidence [36].

We report here a systematic study on the comparison of charge exchange, SID and SIR reactions of doubly charged and singly charged molecular ions SF_4^{2+} and SF_4^+ upon impact on a stainless steel surface at incident energies ranging from a few eV to 60 eV utilizing a tandem mass spectrometer.

2. Experimental

The relative abundance of the product ions as a function of the incident projectile ion energy (collision-energy resolved mass spectra, CERMS curves) was measured with the tandem mass spectrometer apparatus BESTOF described in detail in our earlier papers [8,37]. Projectile ions were produced in a Nier-type electron impact ion source (using 94 eV electrons) operated at pressures of about 10^{-5} Torr. The ions produced were extracted from the ion source region and accelerated to 3 keV for mass and energy analysis by a double-focusing two-sector-field mass spectrometer. After passing the mass spectrometer exit slit, the ions were refocused by an Einzel lens and decelerated to the required incident energy before interacting with the target surface. Shielding the target area with conical shield plates minimized field penetration effects. The incident impact angle of the projectile ions was kept at 45° and the scattering angle (defined as a deflection from the incident beam direction) was fixed at 91°. The incident energy of ions impacting on the surface is defined by the potential difference between the ion source and the surface. The energy spread of the primary ion beam can be determined by measuring the (reflected) total ion signal as a function of surface potential. The energy resolutions (FWHM) of the of primary SF_4^{2+} and SF_4^+ beams were approximately 0.50 and 0.25 eV, while the ion currents were around 30 and 100 nA, respectively, focused on a spot of roughly 2 mm^2. A fraction of the product ions formed at the surface exited the shielded chamber through a 1 mm diameter orifice. The ions were then subjected to a pulsed deflection-and-acceleration field that initiated the time-of-flight analysis of the ions. The second mass analyzer was a linear time-of-flight mass spectrometer with a flight tube of about 80 cm length. The mass selected ions were detected by a double-stage multi-channel plate, connected to a multi-channel scaler (time resolution of 8 ns per channel) and a computer. The product ion intensities were obtained by integration of the recorded signals.

The surface was a polished stainless steel surface at room temperature, covered by background hydrocarbons. Normally, the background pressure of the scattering chamber was better than 10^{-9} Torr when the valve between this chamber and the mass spectrometer was closed. However, during experiments, opening this beam-line valve increases the pressure of the scattering chamber to 2.0×10^{-8} Torr. Under these conditions, the number of collisions between the background molecules and the surface is of the order of 10^{12} mm^{-2} s^{-1}. Given that the ion current was of the order of 10^{11} particles mm^{-2} s^{-1}, it is reasonable to assume that the surface stayed covered with hydrocarbons throughout the experiment duration despite of possible sputtering of the adsorbates by the impinging ion beam.
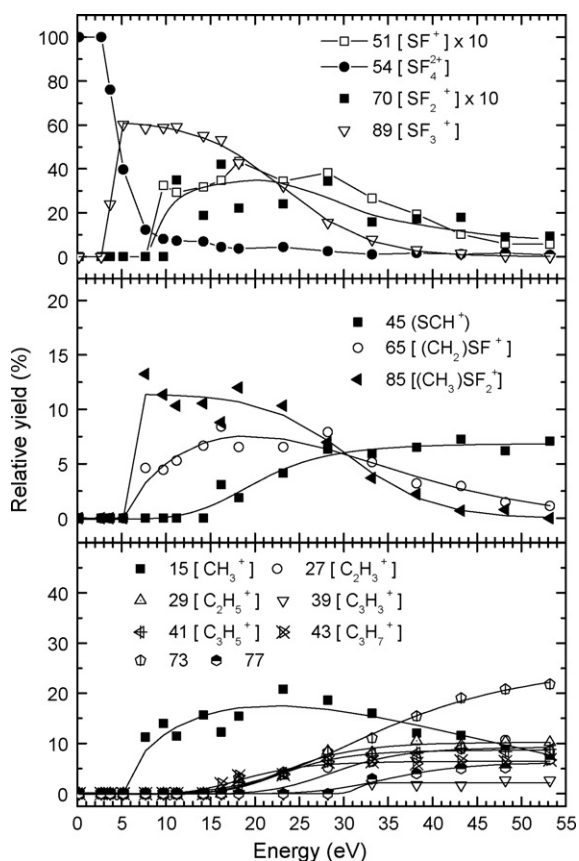

Fig. 3. CERMS for surface-induced reaction of SF_4^{2+} projectiles after impact on a stainless steel surface covered with hydrocarbon layer. The lines between points are only to guide the eye. Top panel: SF_4^{2+} fragments, note that the intensities of masses 51 and 70 are multiplied by 10; Middle panel: chemical reaction products; Lower panel: sputtered compounds.

3. Results and discussion

Fig. 1 shows the primary ion mass spectrum after ionization of SF_6 molecules in the Nier-type ionization source by electron impact of 94 eV electrons at a pressure in the ion source of about 10^{-5} Torr. The most abundant fragment in the spectrum is SF_5^+ $m/z = 127$ and the most abundant doubly charged fragment ion is SF_4^{2+} $m/z = 54$, whose abundance is about 8% of that of SF_5^+. The relatively large electron impact cross-section for the production of the doubly charged ion SF_4^{2+} ([38] and references therein) is presumably due to the formation of SF_4^{2+} by removal of two F^- ions [39].

Fig. 2 shows examples of mass spectra of the product ions scattered from the stainless steel surface (covered with background hydrocarbons) upon impact of doubly charged ions SF_4^{2+} at several collision energies up to 45 eV. Fig. 3 summarizes the respective CERMS curves (plots of the normalized abundance of the product ion $I/\Sigma I$ as a function of the incident energy) of the product ions observed. The CERMS curves of Fig. 3 are divided in three panels for the sake of clarity. The upper panel of Fig. 3 presents the relative yields for the observed fragment ions of SF_4^{2+}, namely SF_3^+, SF_2^+ and SF^+. In the middle panel we present the chemical reaction products $[CH_3SF_2]^+$, $[CH_2SF]^+$, and SCH^+, and in the lower panel we present the sputtering products. The lines joining the data points are arbitrary fits intended only to guide the eye.

The only dication observed in the mass spectra is the scattered incident dication SF_4^{2+}, the relative abundance of which quickly decreases with increasing incident energy. It is interesting to note that neither Coulomb explosion (producing $F^+ + SF_3^+$[29,30]) nor neutral loss (producing SF_3^{2+}[31,32]) processes are observable within our detection limits. This implies that in the present energy range, the capture of at least one electron by this projectile is the major pathway for the dissociative process. Indeed, results from gas-phase experiments suggest that neutral loss from SF_4^{2+} has a
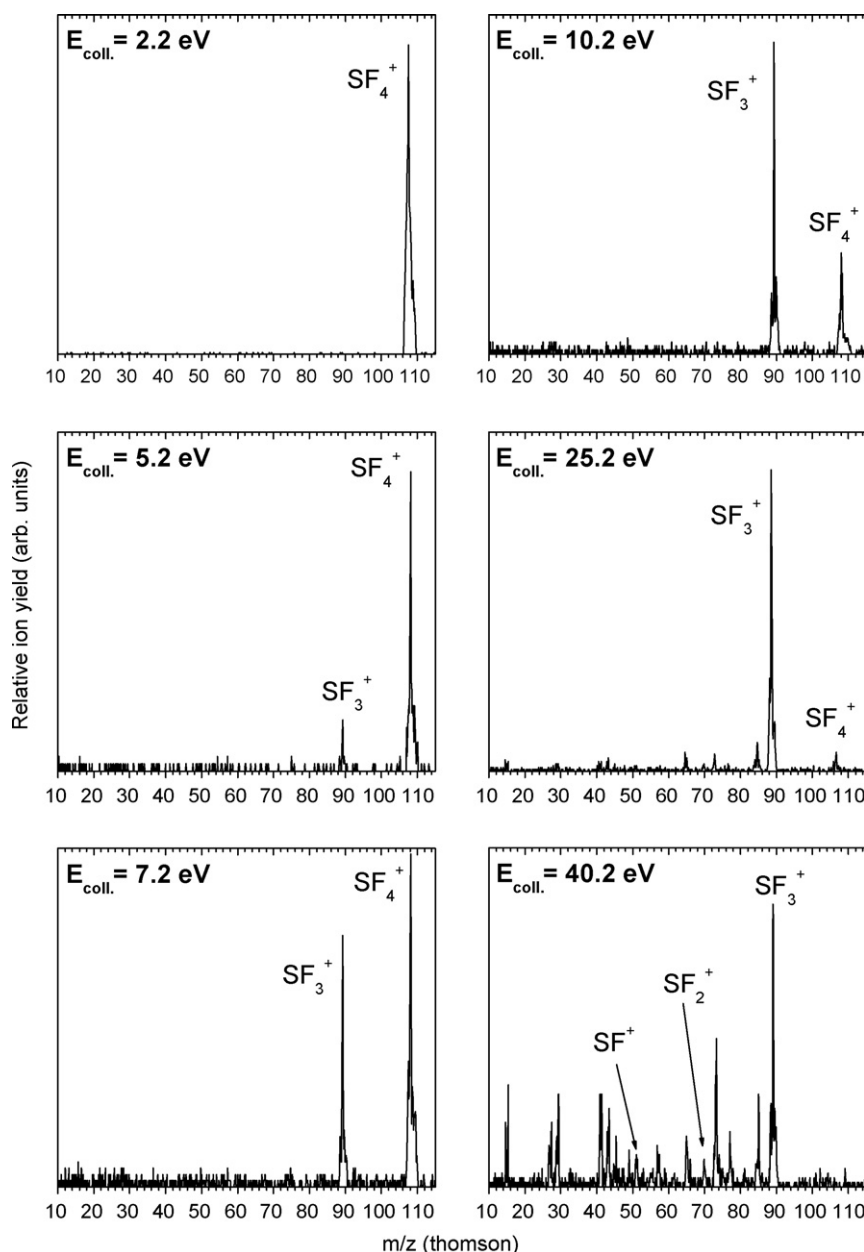

Fig. 4. Product ion mass spectra generated by the surface impact of singly charged ions SF_4^+ at collision energies of 2.2, 5.2, 7.2, 10.2, 25.2 and 40.2 eV.

quite high activation energy, possibly of some eV [32]. The internal energy acquired by a projectile ion due to the collision is well established for this type of surfaces (metal surface covered with hydrocarbons), and amounts only to about 6% of the incident translational energy [40,41,42]. Taking this into account, the energy required for neutral loss in the laboratory frame would be in our case close to the higher part of our energy range, where sputtering (see below) might be hindering the detection of this channel's products.

All other ions detected are singly charged ions. Of them, SF_3^+ (m/z 89), SF_2^+ (m/z 70), and SF^+ (m/z 51) are evidently products of charge transfer at the surface and fragmentation of the internally excited cation SF_4^{+*} formed. The putative charge transfer product SF_4^+ is not observed at all, SF_3^+ is considerably more abundant than the other fragments, and the thresholds of the fragment ions are about 3 eV, 8–10 eV, and 8 eV for the fragment ions SF_3^+, SF_2^+, and SF^+, respectively. The absence of SF_4^+ is consistent with the results from gas-phase collisions with a variety of targets [31] and can be understood in view of the very low energy of the SF_3^+–F bond, i.e., around 0.36 eV [33]. Indeed, this energy should be readily available due to the charge exchange process itself, given that the difference between heats of formation of SF_4^{2+} and SF_4^+ is roughly 20 eV (using [33–35,29,30]), and that the ionization energies of the hydrocarbon adsorbates range between 9.5 to 10 eV [28]. Regarding SF_2^+ and SF^+, their appearance with much smaller yields and at slightly higher energies seems to be consistent with the additional energy requirement for the subsequent F or F_2 abstraction from SF_3^+ after the initial dissociative charge exchange.

The product ions at m/z 85, 65, and 45 are of special interest. These masses can be ascribed to ions $SF_2CH_3^+$, $SFCH_2^+$, and SCH^+ and their origin attributed to chemical reactions of the projectile ions with the hydrocarbons on the surface and further fragmentation of the product ions formed, namely

$$SF_4^{2+} + CH_3-S \rightarrow SF_2CH_3^+ + (F_2-S)^+$$
$$\rightarrow SFCH_2^+(+HF) \rightarrow SCH^+(+HF) \tag{1}$$
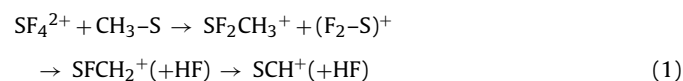
Polyatomic molecular ions are known to react with the surface material. H-atom transfer reactions and formation of a protonated product is a frequent process in collisions of open-shell molecular ions with hydrocarbon-covered surfaces [4]. So far, only pick up of an H-atom or a CH_3 group have been observed [8,12–19,40] or pick up of C_2 units in reactions of fullerene projectile ions with graphite surface covered with background hydrocarbons [43]. In the present case no H-atom pick up has been observed.

In the collision energy range above 40 eV, the spectra are dominated by sputtered hydrocarbons of different $C_nH_x^+$ groups, as well as other compounds at masses 73 and 77. The assignment of these masses as contaminants and not as part of the dissociative reactions was verified by an additional experiment using Ar^+ ions as projectiles in the same experimental setup. It is interesting to note that the behaviour of the CH_3^+ curve with energy resembles more the behaviour of the chemical reaction products than of the other sputtering products. This might be due to CH_3^+ production from break-up of mass 85, but at present our experiment does not allow such conclusion to be drawn, due to the appearance of CH_3^+ even during the Ar^+ experiment (albeit at much higher appearance energy).

From the CERMS curves one observes that the thresholds energies for most of the sputtered species lie around 14–16 eV. This value is remarkably low, but not unprecedented. Vincenti and Cooks [28] had observed similar phenomena with organofluorine ions, with sputtering thresholds as low as 20 eV, but unfortunately their work included only singly charged species. In their analysis the enhanced sputtering is attributed to the charge exchange of the

projectile ions with the surface adsorbates, rather than to a simple kinematic mechanism.

For a better understanding of the dissociation processes of the dication SF_4^{2+}, we performed under identical conditions an equivalent experiment using SF_4^+ ions as projectiles. The results for these additional experiments can be seen in Fig. 4, with corresponding CERMS curves presented in Fig. 5.

The first obvious difference between the CERMS curves for SF_4^{2+} and SF_4^+ is a much smoother dependence of the relative yields of SF_3^+ on collision energy. As a matter of fact, in this case the observed threshold, of 2.4 eV, seems to be a real one, and within error bars consistent with the 0.36 value for the [SF_3^+–F] bond (taking in account once again an average 6% kinetic-to-internal energy transfer during ion collisions with hydrocarbon-covered surfaces). Moreover, the production of SF_2^+ and SF^+ is much diminished in the case of the cation projectile in respect to the dication, and their thresholds energies have risen considerably. The threshold for the [CH_3SF_2]$^+$, [CH_2SF]$^+$, and CHS^+ ions is substantially higher in the case of the singly charged projectile. Moreover, the fitted curves that are used to guide the eye suggest that the threshold for [CH_2SF]$^+$ would be as much as 4 eV higher than the one for [CH_3SF_2]$^+$, and taking into account a 6% energy transfer this would indicate an activation barrier for the HF loss of around 0.24 eV. Despite the fact that the number of experimental points close to the threshold is not sufficient to establish this number with good certainty, the value obtained is clearly once again an upper boundary. Although we could not find in the literature any value for the activation energy
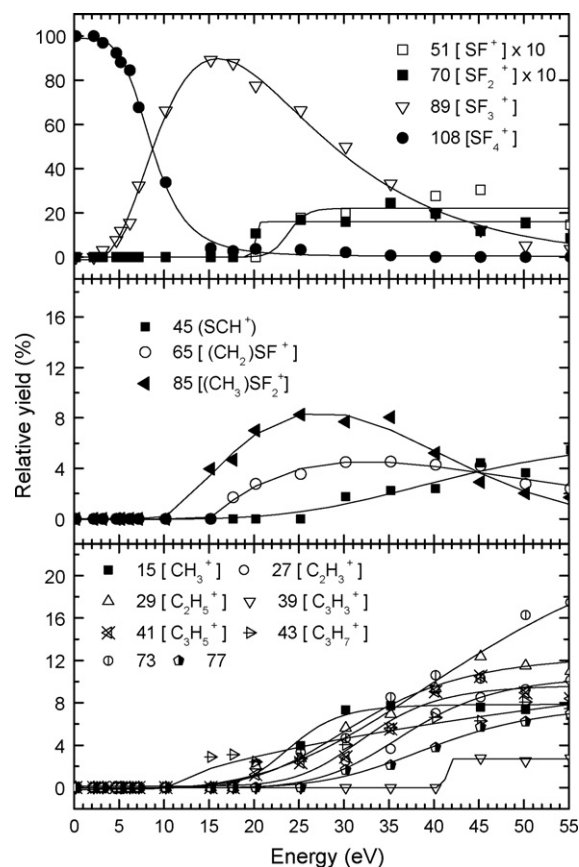

Fig. 5. CERMS for surface-induced reaction of SF_4^+ projectiles after impact on a stainless steel surface covered with hydrocarbon layer. The lines between points are only to guide the eye. Top panel: SF_4^+ fragments, note that the intensities of masses 51 and 70 are multiplied by 10; Middle panel: chemical reaction products; Lower panel: sputtered compounds.

of HF loss from $[CH_3SF_2]^+$, the value of 0.24 eV obtained above seems quite low when compared to other fluorinated species. For $CF_3CH_2CF_3$, for instance, a value of 73 kcal/mol (3.16 eV) [44] has been reported, while for C_2H_5F a value of 49 kcal/mol (2.12 eV) [45]. This observation suggests that the $[CH_3SF_2]^+$ may be formed very reasonably in an internally excited state at threshold.

Regarding the adsorbate sputtering, there is not much difference between the dication and cation cases. It is obvious that the energy released from the single charge exchange of the dication is only available for the channels that involve charged fragments of the projectile molecule, which is not the case for simple sputtering.

4. Conclusions

We have presented data for scattering of SF_4^{2+} and SF_4^+ on a hydrocarbon-covered stainless steel surface. Collision-induced dissociation of the cation was observed while for the dication dissociative charge exchange at the surface and further fragmentation of the cation formed was the major pathway. Both projectiles chemically reacted with the surface adsorbates, producing $[CH_3SF_2]^+$, $[CH_2SF]^+$, and CHS^+ but in the dication case, the single charge exchange reaction provided the outgoing ions with enough energy to overcome reaction thresholds.

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