Partial dissociative ionization of SF_6 by electron impact using an ejected electron-ion coincidence technique

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The relative differential partial ionization cross sections for the production of ionic fragments of SF_6 molecule under impact of 10–20 keV electrons have been measured using an ejected electron-produced ion coincidence technique in a crossed beam apparatus with a time-of-flight analysis of the ions. The detection angle of the ejected electrons of nondiscriminated energies was kept at 90° with respect to the incident electron-beam direction. The 14 ionic fragments: ${SF_5}^+$, ${SF_4}^+$, ${SF_3}^+$, ${SF_2}^+$, SF^+, S^+, F^+, ${SF_4}^{2+}$, ${SF_3}^{2+}$, ${SF_2}^{2+}$, S^{2+}, F^{2+}, ${SF_3}^{3+}$, and S^{3+} resulting from the dissociative ionization of the SF_6 molecule were identified and their relative production cross sections have been measured. The branching ratios of 8 ionic fragments SF_m^{n+} ($m=1$ to 5 and $n=1$ to 3) as a function of impact energy have been determined. These ratios are found to have an almost a constant value over the considered impact energy range within the experimental uncertainty of the measurements. For the investigated impact energies, no previous data or theoretical calculations exist for a direct comparison with the present results.

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I. INTRODUCTION

The electron impact for producing dissociation and ionization of molecules continues to hold much promise for future investigations, particularly when the coincidence studies on multiple charged ions are performed [1]. Despite the fact that long-lived multiple charged molecular ions have been observed in mass spectrometry experiments conducted over the past several decades, studies of such species continue to pose a significant experimental and theoretical challenge [2]. Electron capture by multiple charged molecular ions has been proposed as a mechanism for generating ions in the earth's upper atmosphere [3]. Molecular photoionization has been suggested as a source of energetic charge particles in the terrestrial ionosphere and in the interstellar medium [4]. Leach [5] has considered the formation and destruction of doubly charged polyatomic molecules in interstellar space and has concluded that such species can be found by interaction of molecules with high-energy photons, cosmic ray particles and by energetic shock heated particles.

Owing to the special properties of SF_6 molecule, namely, its high dielectric strength, its chemical inertness and high saturation vapor pressure at a room temperature, it has received a wide applications in industry as a gaseous insulator in high-voltage electrostatic generators, transformers, condensers, and cables [6]. It is also used in plasma etching technique [7]. It is therefore, a matter of primary concern to obtain a thorough understanding of the nature and the properties of the fragmentation product of SF_6 under energetic electron bombardment.

Many workers have studied the SF_6 molecule; partial dissociative ionization cross sections for ${SF_m}^{n+}$ ions with $m=0-5$ and $n=0-4$ and also for F^+ and S^+ ions have been measured [8–15]. Sanderson *et al.* [16] have made the detailed observations of the multielectron dissociative ionization (MEDI) of SF_6 using a time-of-flight (TOF) spectrometer and the 60-fs laser pulses at 750 nm with 7×10^{15}-W cm^{-2} peak intensity. They have been able to make predictions of the major channels of MEDI of SF_6 molecule and to show a good agreement between the peak positions of the measured kinetic-energy distributions assuming the same critical internuclear separation and the calculated kinetic energies of the fragment ions. The coincidence method used by previous workers (see, e.g., Refs. [12,15]), has involved a nondifferential cross sections, that is, total partial ionization cross sections, no information is available in these experiments on the angular and energy distributions of the ejected electrons. However, only recently Al-Nasir *et al.* [17] have measured the partial double-differential cross sections (PDDCS) of the SF_6 molecule by electron impact at relatively low incident energies (100 eV and 200 eV) using an ejected electron-produced ion coincidence technique. The ejected electrons were measured at angles between 10° and 120° with respect to the incidence electron-beam direction, while their ejection energy was varied between 30 eV and 150 eV. At these impact energies, Al-Nasir *et al.* could observe only different fragments of ${SF_m}^{n+}$ molecular ions in their TOF spectra, no atomic ions except S^+ were observed. The well-known interesting features in their experiments, namely, alternation in relative intensity of ${SF_m}^{n+}$ molecular ions with odd-and even-F atoms, as well as the absence of SF_6^+ ion in the TOF spectra were noted and discussed. In the present work, we report on the relative differential partial ionization cross sections (DPICS) of SF_6 molecule by 10–20 keV electron impact using an electron-ion coincidence technique. The ejected electrons of nondiscriminated energies were observed at 90° with respect to the incidence electron-beam direction in coincidence with the produced ions. These measurements are expected to throw some additional infor-

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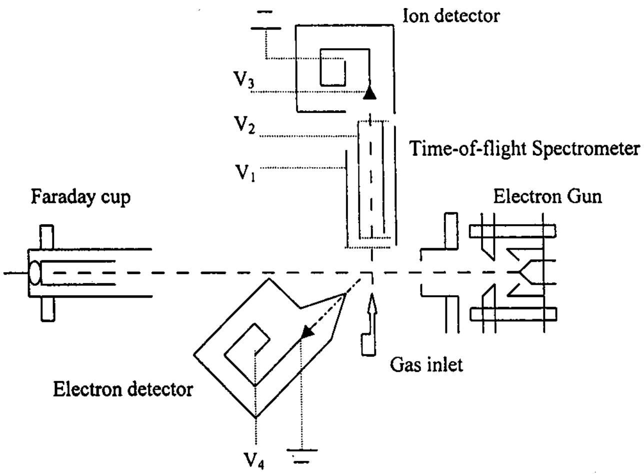

FIG. 1. The schematic diagram of the experimental setup. $V_1 - V_4$: various voltages applied to the ion and the electron detectors.

mation about the ionization mechanism, as well as on the dependence of impact energy upon the branching ratio of different ion fragments of the SF_6 molecule. The results obtained from this work are also qualitatively compared with the results of other workers and with those of photoionization experiments. However, no previous data or theoretical predictions exist at the considered impact energies with which our results can be compared directly.

II. EXPERIMENTAL METHOD

The experimental apparatus used in the present study has been described in detail in our recent publications (see, Ref. [18–20]) and shown in Fig. 1. In brief, a well-focused beam of electrons ($\Phi = 3$ mm, current <1 nA) is made to cross fire a beam of neutral gaseous molecules of SF_6 effusing from a multicapillary tube ($\Phi = 5$ mm) at 90° with each other. From the interaction region, the collisionally induced electrons of nondiscriminated energies ejected at 90° to the incident electron-beam direction are detected by a channel electron multiplier (CEM) with a small solid angle ($\sim 10^{-2}$ Sr). Fragment ions produced in the interaction zone are extracted by an electric field of 275-V cm^{-1} applied between the two condenser plates of the TOF spectrometer. Different charge states of the ions were separated by the TOF spectrometer depending on their mass M to charge q ratios. The required flight times of the ions to travel between their birthplace and the detector (CEM) are proportional to $\sqrt{M/q}$. The cone of CEM of TOF spectrometer was biased at -3.5 kV. Both electrons and ions were monitored on their respective CEMs in a pulse-counting mode. Ion signals derived from the TOF were used as stop signals while those from the electrons as start signals to a time-to-amplitude converter. The details of signal processing and of coincidence circuits are given in Ref. [20]. The time delay of about 3.5 μs between the arrival of ejected electrons and that of simultaneously produced ions at the respective CEMs gives information about the charge states of different ions. A Pentium III computer based 4 K-channels Oxford PCA-3 multichannel analyzer was used for data acquisition, stripping, and plotting, etc.

In the present experiments; the resulting coincidence spectra correspond to the collision events from which the collision-induced target ions of a given charge state and the corresponding electrons ejected at 90° to the incident electron-beam direction are observed simultaneously; such coincidence spectra provide “the correlation possibilities” for the production of a final ion-charge state upon emission of electrons of all energies and having undergone a deflection of 90° with respect to the incident-beam direction. The acceptance angle of the channeltron for detecting the ejected electrons is determined to be $\Delta\theta_{ej} = 1.6°$. These measurements determine the DPICS of the multiple charged ions produced in these energetic collisions. In addition, these measurements are expected to add more detailed information on the collision induced n-fold ionization mechanism than to those which have employed a simple mass spectrometer for determining the abundances of different charged state ions.

The detection efficiency of the ion detector, the transmission efficiency of the TOF spectrometer, and the complete extraction of the recoil ions have been determined in separate experiments and they have been described in our earlier papers [18–20]. The target gas pressure was constantly maintained at 2.4×10^{-4} Torr throughout the experiments to ensure a “single collision condition;” the base pressure of the scattering chamber was obtained at better than 1.5 $\times 10^{-6}$ Torr. The typical count rates of ejected electrons and that of target ions in 16-keV e^--SF_6 collisions were found to be 1200 sec^{-1} and 6000 sec^{-1}, respectively. The data collection times for obtaining the coincidence counts in SF_5^+ peak (the most intense peak) with less than 1% statistics were found to range from 3 h to 5 h in the whole impact energy range of the present measurements. The time resolution $\Delta t/t$ in the present experiments was found to be about 4.4%.

The relative partial ionization cross sections of SF_6 fragment ions are related with the relative ion count rates as, $\sigma^n = S_n / k\mu$, where S_n is the relative count rate of ions in charge state n, k is the over all detection efficiency of detectors, and μ is the effective target thickness of the target (which is directly related to the target gas pressure).

III. MECHANISM OF ION FRAGMENTATION AND DATA ANALYSIS

We have measured the time-of-flight spectra of fragment ions of SF_6 molecule for impact of 10–20 keV electrons with SF_6 using an ejected electron-produced ion coincidence technique. The statistical errors of data collection were generally less than 1% for highly abundant ions but they reached values upto 4–20% for the low-abundant ions. Figure 2 shows a typical time-of-flight spectrum of fragment ions of SF_6 molecule produced by impact of 16.0-keV electrons with SF_6 gas. Different ion fragments of the parent molecule, for example, ${SF_n}^+$ ($n = 1$ to 5) and ${SF_m}^{2+}$ ($m = 1$ to 4) are observed in association with a multiple ionization of the constituent atoms of the molecule. These fragments were identified according to their linear relationships between mass to charge ratios (M/q) and their respective times of flight. The calibration curve for identifying different ion fragments is shown in Fig. 3. It is found that some of the ion

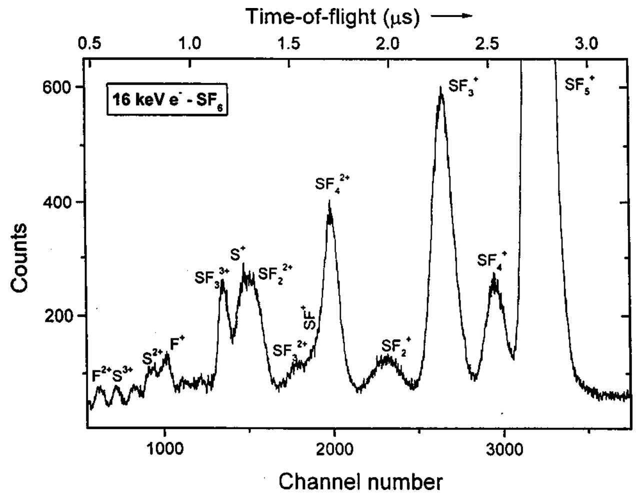

FIG. 2. A typical time-of-flight spectrum of $SF_m{}^{n+}$ ions ($m=0-5$, $n=1-3$) produced in 16.0-keV e^--SF_6 collisions. Spectra are recorded by observing the coincidences between produced ions and simultaneously ejected electrons of indiscriminated energies at 90° with respect to the incident-beam direction, in a single collision condition.

peaks are not clearly resolved in the spectrum; for instance, the ion pairs: $SF_4{}^{2+}$ and SF^+; $SF_2{}^+$ and $SF_5{}^{2+}$; S^+ and $SF_2{}^{2+}$.

The ionic fragmentation of polyatomic molecules occurs mainly via two mechanisms; that is, multielectron-dissociative-ionization (MEDI) and double-dissociative-ionization (DDI). In a MEDI process, many electrons are rapidly removed from the molecule, which is followed by a Coulomb explosion producing the atomic ions of high kinetic energies. Generally, MEDI process takes place in collisions between target molecules and highly intense laser pulses (see, e.g., Ref. [16]), highly charged ion impact [21] and in collisions between high-energy molecular ions and a thin foil of solid targets [22]. Sanderson *et al.* [16] concluded

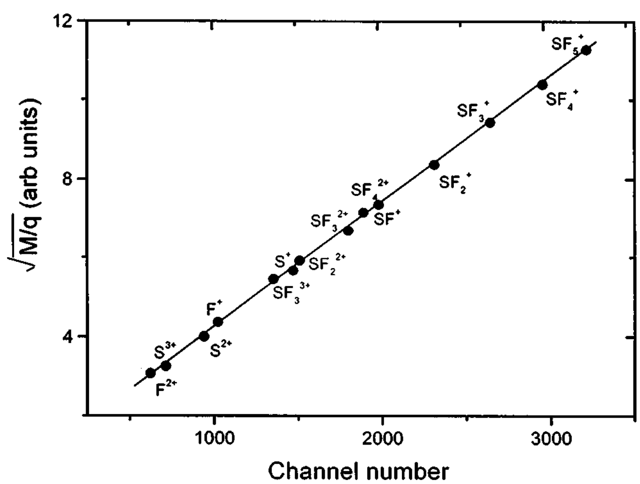

FIG. 3. A calibration curve for identifying different ion peaks in the time-of-flight spectra plotted between $\sqrt{(M/q)}$ and time-of-flight (or channel number).

that the dissociative ionization process caused by electron impact cannot produce the higher stages of ionization resulting into a MEDI process; it rather commonly removes two electrons from the parent molecule, which is responsible for the DDI. In the DDI process, much lower energy is released that cannot dissociate the polyatomic molecules into its constituents atomic ions. However, this is not fully true in case of fragment ions produced by sufficiently energetic electron impact, such as, with the impact energy of our current interest. There is an indication of removal of more than two electrons from constituents of the molecules and we have in fact, observed the multiple ionization of SF_6 molecule yielding $SF_3{}^{3+}$ and the S^{3+} ions (see, Fig. 2); such a high multiple ionization is not expected simply by a DDI process alone.

Commonly, most of the observed ionic fragments of SF_6 molecule by electron impact are produced due to a DDI process (such as shown in Fig. 2). In that case, two electrons from SF_6 are removed and $SF_6{}^{2+}$ ion is formed; however, $SF_6{}^{2+}$ is not observed in the present as well as in the previous works [12,17]. Hence, the SF_6 molecule must be dissociated in different charge state fragments, most probably following two path ways [8,11,12]:

$$SF_6+e_i^- \to SF_6{}^{2+}+2e^- \to SF_m{}^{+}+F^{+}+(n-1)F+2e^- \quad (1)$$

and

$$SF_6+e_i^- \to SF_6{}^{2+}+2e^- \to SF_m{}^{2+}+nF^{+}+2e^-, \quad (2)$$

where, $m+n=6$, e_i^- = incident electrons and e^- = ejected electrons.

Most of the observed singly and doubly charged fragments of SF_6 could be explained with the above dissociative mechanisms. Surprisingly, the singly charged ions of the parent molecule ($SF_6{}^+$) is not present at all in any condition, most probably, it is due to the reason that it dissociates into $SF_5{}^+ + F$ constituents in a very short time of the order of picosecond [16]. Another interesting feature is observed in the ionic fragmentation of SF_6 molecule; that is, a periodic alternation of the relative intensities of ions with different values of charge states. The singly charged fragments, SF_n^+ having odd values of n are found to have higher intensities than those of even value of n. On the other hand, the doubly charged ionic fragments, $SF_m{}^{2+}$ having even values of m, are observed with higher intensities than those of odd value of m. A similar behavior of alternation of intensities has been also observed previously by Hitchcock, Brion, and Van der Wiel [23] and Al-Nasir *et al.* [17] by electron impact and by Hitchcock and Van der Wiel [12], Masuoka and Samson [13], and Sanderson *et al.* [16], by photon impact. This intensity pattern could be explained with the simple valence bond description of the different ion fragments and with hybridization mechanism [12].

The ground electronic configuration of sulfur is $1s^2\,2s^2\,2p^6\,3s^2\,3p^4$; combination of two d-, one s-, and three p-orbitals does give six equivalent d^2sp^3 hybrid orbitals and thus constituting the geometry of SF_6 molecule in the ground state. The sulfur-fluorine bond in SF_6 is not particularly strong, that is, SF_5—F bonding energy is less than 3.4 eV [24]. Due to the symmetrical configuration of SF_6 (a regular octahedral), ${SF_6}^+$ is highly unstable and exhibits Jahn-Teller instability [25] as suggested by Dibeler and Walker [9]. Therefore, due to this extreme instability, the most probable dissociation process of ${SF_6}^+$ ion is a loss of one fluorine atom from it and producing a ${SF_5}^+$ ion. However, some workers [11,26] have reported the existence of SF_6^+ ion with a very small intensity.

Most of the singly charged fragments of SF_6 molecule are considered to be constructed from the valence configuration of S^+, i.e., $3s^2\,3p^3$ and the appropriate number of fluorine atoms. Excitation of one $3s^2$ electron of S^+ into $3d$ orbital causes dsp^3 hybridization, which in turn provides five equivalent orbitals to combine with five fluorine atoms. This stable configuration explains the stability of SF_5^+ ions. The ground-state configuration of S^+ that has a half filled $3p$ sub shell, i.e., $3p^3$ also prefers the bonding with three fluorine atoms and thus explaining the observed stability of SF_3^+ ions. On the other hand, no such stable configuration can be derived for ${SF_2}^+$ and ${SF_4}^+$ ions with the half filled $3p$ configuration; therefore, these latter fragments are expected to be much less intense. Similarly, the doubly charged fragments of SF_6 molecule are considered to be formed from a valence configuration of S^{2+}, i.e., $3s^2 3p^2$. In this configuration, the most favorable situation is where one electron from $3s$ sub shell goes to $3p$ sub shell and a sp^3 hybridization is established; which in turn provides four equivalent orbitals to combine with four F atoms. This explains the relatively high stability of ${SF_4}^{2+}$ ions. The S^{2+} configuration also prefers to combine with two fluorine atoms in absence of *sp* hybridization. This has explained the predicted intensity of ${SF_2}^{2+}$ ions. However, no such type of configuration is derived for ${SF_m}^{2+}$, having odd value of m. Furthermore, we observe the ${SF_3}^{3+}$ ion having a relatively higher intensity than ${SF_3}^{2+}$ ion. This relative stable configuration of ${SF_3}^{3+}$ could be explained in terms of valence configuration of S^{3+} (i.e., $3s^2 3p^1$), followed by sp^2 hybridization. A relatively weaker peak seen in between S^{2+} and S^{3+} peaks could not be assigned to any ionic fragment.

As mentioned earlier, many peaks in the time-of-flight spectrum of SF_6 molecular ions are found to overlap one over the other [see, Figs. 2 and 4(a)], it was, therefore necessary to fit the peaks with a multipeak Gaussian fitting program for calculating the net areas of individual peaks. The observed TOF spectra of the SF_6 ionic-fragments produced in 10-keV e^--SF_6 collisions and the corresponding peak fitted patterns of the resulting spectrum after background subtraction are shown in Figs. 4(a) and 4(b), respectively. Details of the peak intensities and the branching ratios of the various ionic fragments of SF_6 are discussed in the following section.

IV. RESULTS AND DISCUSSION

The typical TOF spectra of fragment ions of SF_6 molecule recorded for 16-keV and 10-keV electron impacts using an ejected electrons-produced ions coincidence technique are

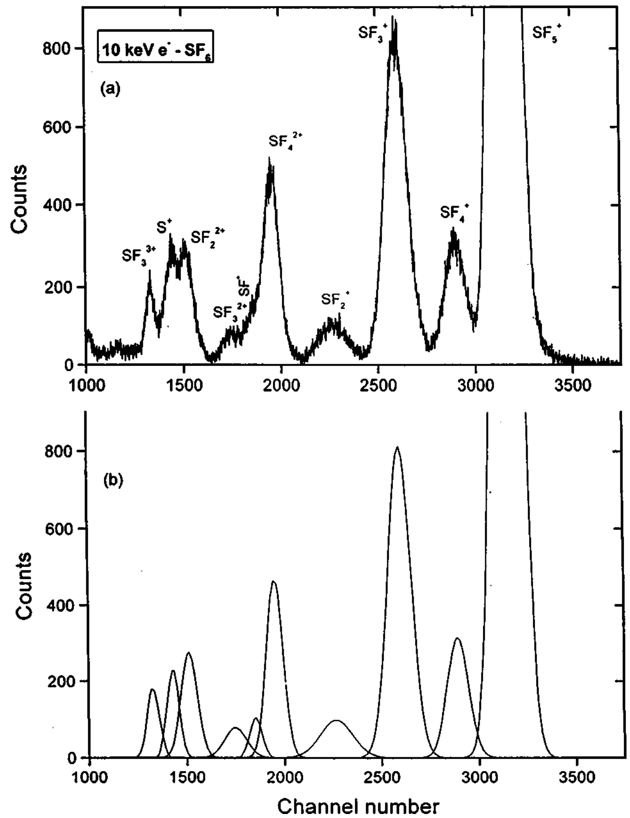

FIG. 4. Time-of-flight spectrum of the SF_6 ionic-fragments produced in 10-keV e^--SF_6 collisions (a). The corresponding multipeak Gaussian fitted patterns of the spectrum after background subtraction (b).

displayed in Figs. 2 and 4(a), respectively. The first two basic features as explained in the preceding section are noted: (i) no ${SF_6}^+$ ions are observed, while the most massive fragment ion ${SF_5}^+$ is observed with highest intensity, (ii) an alternation in relative intensities of fragment ions is observed for ${SF_n}^+$ having odd values of n with higher intensities than those of even value of n. Also, the fragments ions ${SF_m}^{2+}$ having even value of m are seen to have higher intensities than those of odd value of m. The third basic feature which has been noted in our measurements is that the multiple charged atomic ions of SF_6 molecule begin to form for electron impact energies larger than 12 keV. The rest of the features as described above remain the same throughout up to 20 keV impact energy. This last feature suggests that the ionization of SF_6 molecule proceeds via both DDI as well as MEDI processes simultaneously for the impact energy greater than 12 keV. This means, the collision events taking place at the impact energies larger than 12 keV release energy so much that more than two electrons are removed from the constituents of the target molecule. The signature of S^{3+} ion peaks attests the process of multielectron ionization mechanism.

Figure 5, shows the relative intensity distributions of different ion fragments of SF_6 molecules produced by 16-keV electron impact. Similar structure has also been observed by other earlier workers [8–15], but the relative intensities of

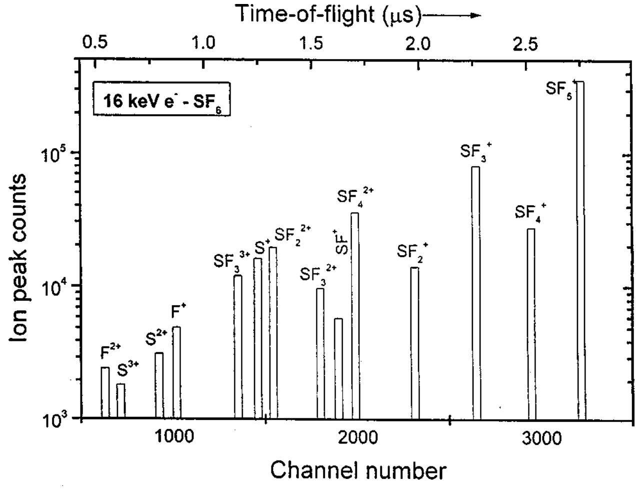

FIG. 5. Relative intensity distributions of different ion fragments of SF_6 molecules produced by 16-keV electron impact, with SF_6 gas.

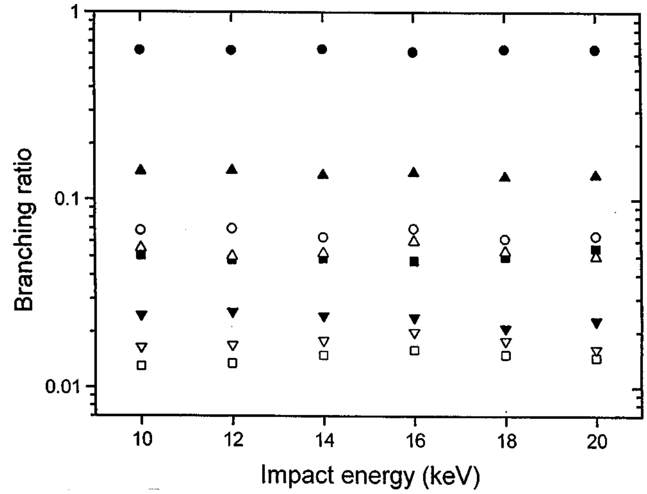

FIG. 6. Impact energy dependence of the branching ratio (fractional ion yield) for different ionic fragments of SF_6 molecule. Present data, ●: ${SF_5}^+$; ■: ${SF_4}^+$; ▲: ${SF_3}^+$; ▼: ${SF_2}^+$; ○: ${SF_4}^{2+}$ $+SF^+$; □: ${SF_3}^{2+}$; △: ${SF_2}^{2+}+S^+$, and ▽: ${SF_3}^{3+}$.

different ion fragments are found to vary from one worker to the other; the reason being the experimental conditions are not the same. We could not directly compare our present results with other experimental data, because in our knowledge, no experimental results of partial dissociative ionization cross sections of SF_6 are available at electron impact energies of the present work. However, if we compare the present results with other electron impact data on a qualitative basis, for instance, with those of Al-Nasir *et al.* [17] and Hitchcock and Van der Wiel [12]. A reasonably strong peak of ${SF_3}^{3+}$ ion is observed for the first time in our all spectra. No such peak was reported earlier both in the work of electron and photon impacts with SF_6. Further, ${SF_5}^{2+}$ ion peak in our work is seen to be mixed with ${SF_2}^+$ ion peak but ${SF_5}^{2+}$ is more pronounced and well separated in the work of Ref. [17]; no signature of the latter ion peak is present in the TOF spectrum of SF_6 ion fragments by a photon impact reported by Sanderson *et al.* [16]. Generally, the similar structure of ion fragments of SF_6 is observed in case of photon impact data containing ions from ${SF_5}^+$ to S^+ (relative intensities are, however different). It is expected that in both cases of electron and photon impacts, most of the fragments are produced through the DDI process. However, in the case of multiple charged states of constituent atoms, these two results are entirely different. Sanderson *et al.* [16] reported that the multiple charged ions of S and F are observed with very high abundances and that the intensity of S^{2+}, S^{3+}, and F^{2+} are higher than intensity of ${SF_5}^+$. They explained this situation in terms of MEDI mechanism. However, in the present results, there is an indication of removal of more than two electrons from one of the constituent atoms of the parent molecule, but its probability is found to be considerably small in comparison to ${SF_5}^+$ ion.

The fractional yields (branching ratios) as the function of impact energy have been determined for different ionic fragments of SF_6 molecule and are shown in Fig. 6. The branching ratio is defined as the ratio of the number of fragment ions of the type X_j to the total number of ions produced $(=\Sigma_j X_j)$. It is seen that the values of branching ratios are constant within the energy range of the present investigation. The fluctuations observed in data points of Fig. 6 for low-abundant fragments are due to the statistical errors and fitting uncertainties. The total uncertainty in the branching ratio is estimated to be less than 3% but it may go over to 10% for low-intensity fragments. A more detailed information about the ionic-fragmentation mechanism involved in these collisions is expected to be derived from the measurements of PDDCS as has been obtained by Al-Nasir *et al.* [17] at relatively very low impact energies. The work on PDDCS measurements of light polyatomic molecules is in progress in our laboratory.

V. CONCLUSIONS

The relative differential partial ionization cross sections of SF_6 molecule by 10–20-keV electron impact using an ejected electron-produced ion coincidence technique have been measured. The ejected electrons of nondiscriminated energies were observed at 90° to the incident electron-beam direction in coincidence with the target ions. The fragments ions: ${SF_5}^+$, ${SF_4}^+$, ${SF_3}^+$, ${SF_2}^+$, SF^+, S^+, F^+, ${SF_4}^{2+}$, ${SF_3}^{2+}$, ${SF_2}^{2+}$, S^{2+}, F^{2+}, ${SF_3}^{3+}$, and S^{3+} have been identified in the recorded time-of-flight spectra having a time resolution of about 4.4%. The above fragments are found to result mainly from the double-dissociative-ionization process; however, the S^{3+} ions are believed to arise from a multielectron dissociative process in the considered energetic electron collisions with SF_6 molecule. The present results confirm the observations of other workers who obtained the ionic fragmentation of SF_6 molecule by electron and photon impact. In our work too, no evidence has been found for the existence of ${SF_6}^+$ ion but the alternation in the intensity of odd and even number of fluorine atoms in singly and doubly charged fragment ions of SF_6 molecule has been confirmed.

In comparison with the results obtained from experiments of photon impact with SF_6 molecule, a remarkably different intensity pattern of various ion fragments has been noted. A reasonably strong peak of ${SF_3}^{3+}$ ion is observed for the first time in our all spectra which was not reported earlier both in the work of electron and photon impacts with SF_6. Further, the branching ratios of 8 ionic fragments: ${SF_m}^{n+}$ ($m=1$ to 5; $n=1$ to 3) as a function of impact energy in the range of 10–20 keV have been measured. The branching ratios are found to exhibit an almost a constant value over the impact energy range of our interest. The measurements of partial double-differential cross sections of SF_6 molecule by 10–30 keV electron impact are presently in progress.

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