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Cluster-assisted generation of multicharged ions in nanosecond laser ionization of carbon bisulfide clusters at 1064 nm*

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The photoionization of seeded carbon bisulfide molecular beam by a 1064 nm nanosecond Nd-YAG laser with intensities varying from 0.8×10^{11} to 5.6×10^{11} W/cm^2 have been studied by time-of-flight mass spectrometry. Multiply charged ions of S^{q+} (q = 2–6) and C^{q+} (q = 2–4) with kinetic energy of hundreds of electron volts have been observed, and there are strong experimental evidences indicating that those multicharged ions originate from the ionization of CS_2 neat clusters in the beam. An electron recolliding ionization model is proposed to explain the appearance of those multiply charged atomic ions under such low laser intensities.

Keywords: carbon bisulfide, multicharged ions, cluster, coulomb explosion
PACC: 3280

1. Introduction

CS_2, as a simple linear symmetric molecule, is one of the most extensively studied molecules by laser spectroscopy and mass spectrometry.[1–12] Patsilinakou *et al*[1] studied the photofragmentation of excited states of CS_2 and CS_2 clusters initiated by multiphoton excitation in a wavelength range from 329 to 345 nm. Four major species, CS^+, CS_2^+, $(CS)_2^+$ and CS_3^+, were observed in their experiments, and the relative abundances of these ions strongly depended on the wavelength. Seaver *et al*[5] measured the multiphoton ionization (MPI) and fragmentation of CS_2 by using lasers at wavelengths of 266 nm and 193 nm with intensities of 10^{11} and 10^{10} W/cm^2, respectively, and observed CS_2^+, S_2^+, CS^+ and S^+ ions by using both lasers. And they also studied the ion signal intensity as a function of laser intensity and reagent pressure and confirmed that the mechanisms involved at these two wavelengths were different.

Recent investigation of molecules in intense laser fields generated by ultrashort high-power laser radiation has revealed a variety of phenomena, such as multiple ionization and Coulomb explosion (CE). Particularly, using picosecond and femtosecond lasers with intensities in a range of 10^{14}–10^{16} W/cm^2, the electric fields generated are no longer small compared with the binding molecular fields of the valence electrons. Hence new physical effects are expected to occur.[13,14] Iwasaki *et al*[6] measured the time-of-fight (TOF) mass spectrum when CS_2 was exposed to the circularly polarized ultrashort laser field (795 nm, 100 fs, 5.5×10^{14} W/cm^2). Abundances of multicharged ions were observed, e.g. CS_2^{z+} (z = 1–3), fragment atomic ions S^{q+} (q = 1–4) and C^{q+} (q = 1, 2). Wu *et al*[10] studied the influences of laser polarization and intensity on the interaction of a 810 nm femtosecond laser with CS_2 molecule, the major ions observed in their experiment were the parent molecular ions (CS_2^+, CS_2^{2+}), singly fragment ions (S^+, C^+), and highly charged ions (S^{q+}, q = 2–5; C^{q+}, q = 2, 3). They believed that field ionization was responsible for the formation of the molecular ions at laser intensities varying from 7.2×10^{13} to 2.2×10^{15} W/cm^2. Graham *et al*[12] studied the ionization and dissociation of CS_2 molecule in an intense linearly polarized laser field of femtosecond pulse duration. The angular distributions of CS_2^{2+}, CS_2^{3+}, CS^+, CS^{2+}, S^{q+} ($q \le 6$)

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and $C^{q+}(q \leq 4)$ arising from the CE of the parent ions were presented at a laser intensity of 1×10^{16} W/cm^2 with a wavelength of 790 nm and pulse duration of 50 fs.

In our recent studies, we observed multicharged ions from the molecule clusters such as benzene,[15,16] methyl iodide[17] and ammonia,[18] irradiated by a nanosecond laser with low intensities in a range of 10^9–10^{11} W/cm^2. In the present paper, we study the laser-initiated ionization of seeded molecular beam of CS_2 by using TOF mass spectrometry with a nanosecond commercial Nd-YAG laser. The purposes of our study are to ascertain whether the cluster-assisted multiple ionization can occur in a simple triatomic molecule CS_2 or not, what kind of multicharged species will form in the process, and how the cluster size and laser parameter, i.e. intensity and polarization, affect the distribution of charge states and kinetic energy of multicharged ions.

2. Experiment

The experimental apparatus used in this study was a linear Wiley–Mclaren two-stage time-of-flight mass spectrometer (TOFMS), which has been described in detail elsewhere.[15−20] Briefly, when a supersonic jet beam was used, the liquid CS_2 bubbled by helium gas of 0.3 MPa was introduced into a source chamber through a pulsed valve (ϕ0.5 mm) and then into an ionization chamber through a skimmer (ϕ3 mm). The pressure in the ionization chamber was about several 10^{-4} Pa at a sample inlet frequency of 10 Hz. The distance from the valve to the ionization point was about 110 mm. When the effused inlet beam was used, the neat CS_2 vapour at room temperature was inlet through a needle valve instead of a pulsed valve, and the pressure in the ionization chamber was kept at 4×10^{-4} Pa. The laser system was a commercial Nd-YAG laser (Spectron SL803, 500 mJ/25 ns at 1064 nm). The intensities of laser at a focus point used in the experiment were in a range from 0.8×10^{11} to 5.6×10^{11} W/cm^2 by adjusting the voltages of the pumping xenon lamp. The laser polarization direction was perpendicular to the drift tube axis and could be rotated to an arbitrary angle by a half-wave plate. The produced ions were first accelerated by the extraction electric field and then detected by a dual microchannel plates (MCP) after 500 mm field-free flight. The total voltage on the extraction plates was 2800 V. The ions were recorded by a digital oscilloscope (Tektronix, TDS224) and averaged over 128 laser shots. The CS_2 purchased from the Shanghai Chemical Reagent Corporation, was of an analytical purity, and it was not further purified in use. The helium gas obtained from the Nanjing Special Gases Corporation had a purity of 99.99%.

3. Results

3.1. Laser ionization mass spectra of CS_2 beam seeded in He gas

Figures 1(a) and (b) show the mass spectra obtained by a 1064 nm nanosecond laser irradiating the effused CS_2 vapour and pulsed helium seeded CS_2 beam, respectively.

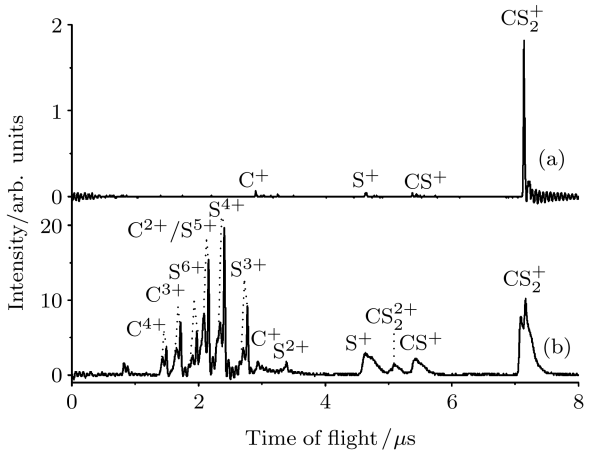


Fig.1. Time-of-flight mass spectra of CS_2 generated by a 25 ns, 1064 nm laser irradiating (a) an effused CS_2 beam and (b) a pulsed beam using 0.3 MPa helium as carrier gas. The laser intensities are (a) 4.8×10^{11} and (b) 1.8×10^{11} W/cm^2.

In Fig.1(a), when the CS_2 molecules were ionized by the laser, the domain ions in the mass spectrum were C^+, S^+, CS^+ and CS_2^+ ions, which are typical fragment ions in multiphoton ionization dissociation (MPID) processes of CS_2 molecules.[21] While the pulsed beam was ionized by a 1064 nm laser at an intensity even smaller i.e. 1.8×10^{11} W/cm^2 as shown in Fig.1(b), many multicharged ions, such as C^{4+}, C^{3+}, C^{2+}, S^{6+}, S^{5+}, S^{4+}, S^{3+}, S^{2+} and CS_2^{2+} ions were observed, and the total intensity of multicharged ions was much stronger than that of singly charged ions (CS_2^+, CS^+ and S^+ ions). The intensities of singly charged ions, CS_2^+, CS^+ and S^+ ions, in the beam are about tens of times stronger than

those in effused beam, and the mass spectra exhibit peak broadening, which is probably due to the space charge effect. With the exception of the peak of the S^{2+}, the peaks of these multiply charged atomic ions split into two parts, corresponding to the forward and backward ejections of the fragment ions, which manifests that these atomic ions were produced through Coulomb explosion.

By measuring the time differences of the split peaks in the mass spectrum, we can estimate the kinetic energies (KEs) of these ions using the following equation:[10,22]

$$E_{\mathrm{k}} = \frac{(U_1 - U_2)^2}{8md^2} q^2 \Delta t^2 = \frac{U_1^2 (1-K)^2}{8md^2} q^2 \Delta t^2, \quad (1)$$

where U_1 and U_2 are the voltages on the repeller and acceleration plates, d is the distance between these plates, K is equal to U_2/U_1, q is the charge of the ion, and Δt is the difference in arrival time between forward and backward ejected ions. In this experiment, the voltage on the repeller, U_1, is 2800 V, K is 0.5 and d is 20 mm. The splitting times of C^{4+}, C^{3+}, S^{6+}, S^{5+}, S^{4+}, S^{3+} are 0.059, 0.063, 0.071, 0.068, 0.065 and 0.066 μs, respectively, and the calculated KEs are 272, 175, 333, 212, 124 and 72 eV, respectively. It should be noted that the peak of CS_2^+ also has a doublet feature. The released kinetic energy of CS_2^+ is about 0.6 eV from the above formula, indicating that the CS_2^+ does not originate from the direct CE process.

3.2. Mass spectra at different parts of the pulsed CS_2 molecular beam

Since the multiply charged atomic ions appear only when the pulsed seeded beam is irradiated by the nanosecond laser other than the effused beam, we think that the generation of multiply charged atomic ions may be attributed to the appearance of the clusters in the seeded beam. The concentration and size of the clusters both vary at different parts of the pulsed beam, and the size is bigger and the concentration of the clusters is higher at the densest part of the molecular beam. We recorded a series of TOF mass spectra at different parts of the pulsed beam by changing the delay of the laser pulse with respect to the opening of the pulsed valve as shown in Fig.2. When the laser interacts with the head of the pulsed beam, the delay time is 520 μs, where the density of clusters is low, we can see that some weak signals of multicharged ions appear in the mass spectrum. The intensities of multicharged ions increase quickly when the laser irradiates the denser part of the molecular beam by increasing the delay time. The intensity of multiply charged atomic ions reaches a maximum, with a delay time being about 820 μs, where the laser interacts with the middle part of pulsed beam and the concentration of the CS_2 cluster is the highest. Keep on increasing the delay time, the intensity of multiply charged atomic ions decreases rapidly, and multiply charged atomic ions do not appear when the delay time exceeds 1120 μs, and the laser interacts with the tail of the pulsed beam where the intensity of the cluster is much lower. All those results about the time delay show that the appearance of the multiply charged atomic ions is closely related to the intensity of the CS_2 cluster.

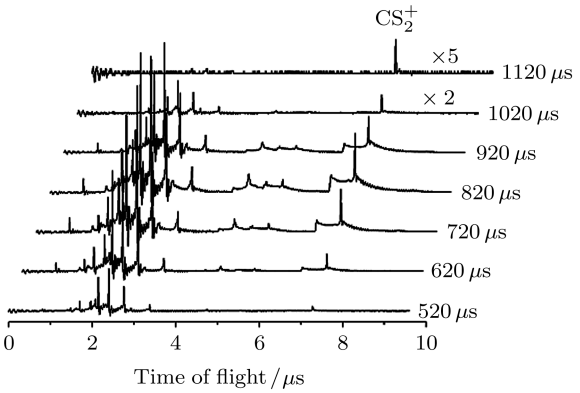


Fig.2. Time-of-flight mass spectra obtained by a laser irradiating different parts of a pulsed seeded CS_2 beam. The time marked on the curves refer to the delay time between the opening time of the pulsed valve and the pulsed laser. The laser intensity is 4.8×10^{11} W/cm^2. Multicharged ions appear only when the delay time is less than 1020 μs and the laser irradiates the densest part of the beam, where the size of cluster is larger and its concentration is high.

3.3. Mass spectra at different backing pressures of helium carrier gas

Figures 3(a)–(e) show the mass spectra of seeded CS_2 beam at different helium backing pressures ranging from 0.2 to 0.6 MPa, the delay time between the opening of the valve and the laser is set to be 820 μs. We can see that the intensities of multicharged ions, C^{q+} (q = 3, 4), S^{q+}(q = 3, 4, 5, 6) increase only slightly with the backing pressure of helium increasing, while the intensities of single charged ions, CS_2^+, CS^+, S^+ and C^+ increase apparently with the increase of backing pressure. The singly charged carrier gas ions, He^+, start to appear when the backing pressure of helium is higher than 0.3 MPa as shown in Fig.3(c),

and its intensity increases almost linearly with the backing pressure increasing.

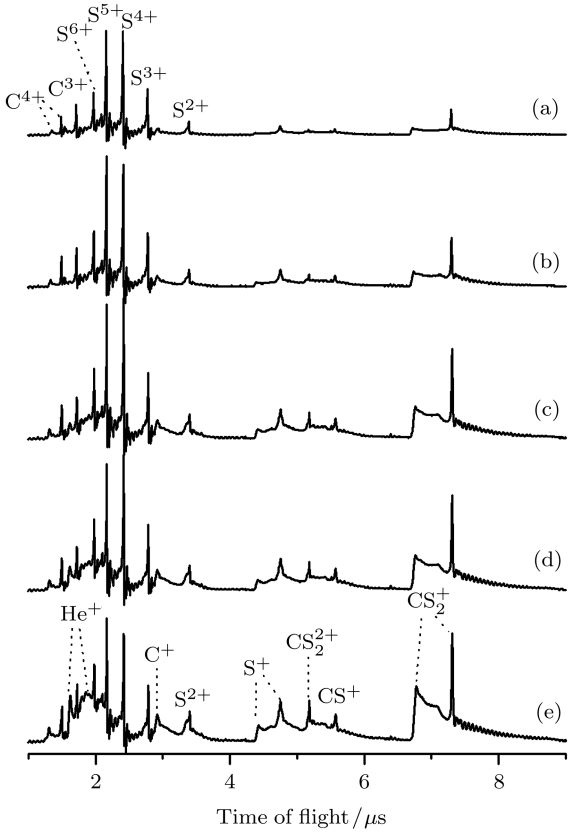


Fig.3. Time-of-fight mass spectra of seeded CS_2 beam at different backing pressures of helium carrier gas: (a) 0.2, (b) 0.3, (c) 0.4, (d) 0.5 and (e) 0.6 MPa. The laser intensity is 5×10^{11} W/cm^2.

The peak shape of a specific m/q often contains the information on the distributions of initial positions and velocities. By changing the ratio, $K = U_2/U_1$, of the electric voltages on the repeller plate (U_1) and extraction gird (U_2) of the two stages of TOFMS, the decoupling of the position information and the velocity information of the product ions may be realized. Figure 4(a)–(d) show some typical mass spectra taken at K = 0.8, 0.67, 0.5 and 0.33, respectively. The peaks of singly charged ions CS_2^+, CS^+, S^+ and C^+ spectra are sharp under the space-focusing condition as shown in Fig.4(a), but quickly become broader as shown in Figs.4(b)–(d), when the ratio is set further out of space-focusing condition by increasing the electric field strength, which implies that these ions have a wide spatial distribution in the extraction region when they are formed. In contrast, the peak profiles of multicharged ions, C^{q+} (q = 3, 4), S^{q+}(q = 3, 4, 5, 6), are the broadest in Fig.4(a), and the peaks split into forward and backward peaks. The splitting is reduced when a higher extraction field is used by reducing the voltage on the extraction grid (U_2) as shown in Figs.4(b)–(d), which suggests that these ions have a very large kinetic energy before being accelerated and their spatial distribution is very narrow in the extraction region.

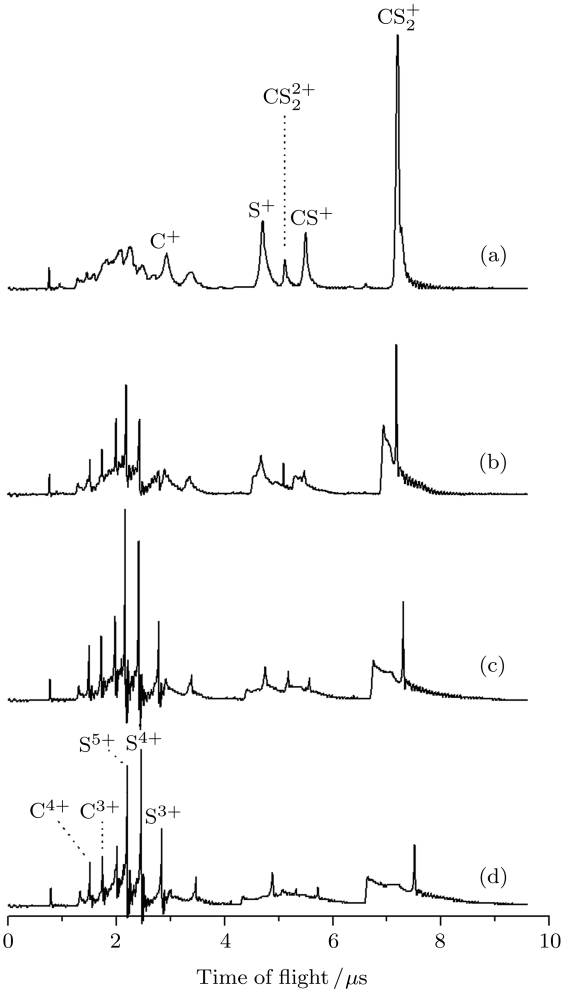


Fig.4. Time-of-flight mass spectra of seeded CS_2 beam at different values of U_2/U_1. The total voltage on the repeller plate U_1 is fixed to 2800 V, the values of K, i.e. U_2/U_1 being (a) 0.8, which is the space-focusing condition of this time-of-flight mass spectrometer, (b) 0.67, (c) 0.5 and (d) 0.33. The laser intensity is 4.8×10^{11} W/cm^2. The strength of electric field in the extraction region, $E_1 = U_1(1 - K)/d$, increases from (a) to (d).

3.4. Mass spectra under different laser powers and polarizations

Figure 5 shows the peaks of multiply charged atomic ions under laser intensities ranging from

0.8×10^{11} W/cm^2 to 5.6×10^{11} W/cm^2. The multicharged atomic ions, e.g. C^{4+} and S^{5+} appear even when the laser intensity is as low as 0.8×10^{11} W/cm^2. The intensity and the split time of the ions increase with the increase of laser intensity, e.g. the ion intensity increases about 500 times from Fig.5(e) to (a), but the charge state distribution does not apparently change.

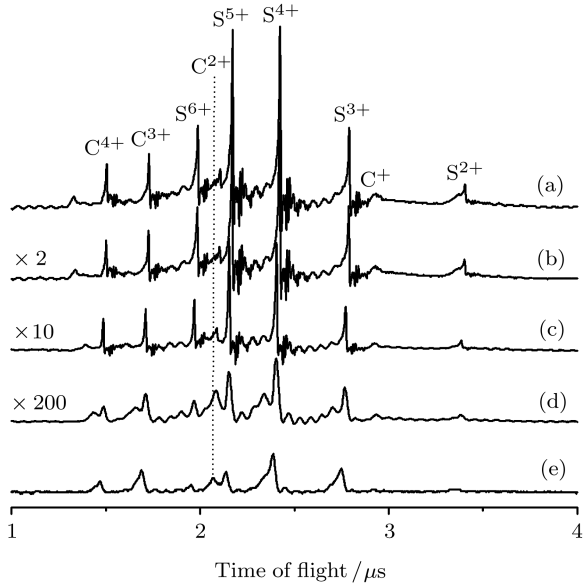


Fig.5. Time-of-fight mass spectra of seeded CS_2 beam ionized by a 1064 nm nanosecond laser with powers: (a) 5.6×10^{11}, (b) 4.8×10^{11}, (c) 2.0×10^{11}, (d) 1.5×10^{11} and (e) 0.8×10^{11} W/cm^2.

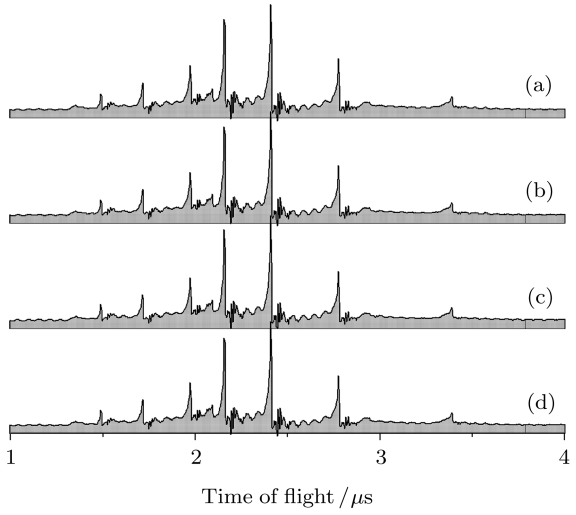


Fig.6. Time-of-flight mass spectra of seeded CS_2 beam ionized by a 1064 nm nanosecond laser in laser polarization directions being (a) parallel to the drift tube axis, (b) at 30° to the drift tube axis, (c) at 60° to the drift tube axis, and (d) perpendicular to the drift tube axis.

Another series of experiments focuses on the effect of laser polarization on the generation of the multiply charged atomic ions. Figure 6 shows the information on typical laser polarization for the multiply charged atomic ions. The angle as shown in Fig.6 corresponds to the angle between the laser polarization and the drift tube axis. When the angle changes from 0° to 30°, 60° and 90°, the multiply charged atomic ions do not change obviously. The fragment ions exhibit isotropic distributions with respect to the laser polarization direction.

4. Discussion

To elucidate the mechanism of the generation of the multiply charged atomic ions herein, we propose several processes that occur on the time scale of the laser pulse.[15−20] These include the initial ionization, the cluster heating and further ionization and the cluster expansion. The first question we need to consider is whether the initial ionization is produced by multiphoton ionization or field ionization. In an intense laser field, the multiphoton ionization and field ionization compete with each other, which may be judged by using the following Keldysh parameter:[23]

$$\gamma = \omega_0 \frac{\sqrt{2E_{\mathrm{IP}} m_{\mathrm{e}}}}{eE_0}, \tag{2}$$

where ω_0 is the laser frequency, E_{IP} is the ionization potential of the molecule (10.07 eV for CS_2), m_{e} and e are the mass and charge of an electron, respectively, E_0 is the electric field strength of the laser.

If $\gamma \gg 1$, the system is in the MPI regime. If $\gamma < 1$, the system is in the field ionization. In our experiment, the maximum laser intensity is about 4.8×10^{11} W/cm^2 and the value of γ is about 10. So the initial ionization is mainly due to the MPI. Some of the electrons generated during the MPI can escape from the cluster, leaving the cluster positively charged, but most of the MPI-induced electrons can be trapped in the cluster due to (a) the rebound from the neighbouring solid-dense neutral atoms of molecules, and (b) Coulomb attraction of the ion core. The trapped electrons are heated by the laser field via inverse bremsstrahlung (IBS) absorption when they collide with the neutrals or ions.[24−27]

The heating rate of the electron (ignoring the energy loss in collision) can be estimated from the fol-

lowing equation:[24,25]

$$\frac{\mathrm{d}\varepsilon}{\mathrm{d}t} = \Delta E \times v, \tag{3}$$

where ε is the mean electron kinetic energy, ΔE is the average energy absorbed by an electron in a single collision with ion or neutral atom and molecule, ν is the frequency of electron collision with neutrals and/or ions.

$$\Delta E \sim U_{\mathrm{P}} = 9.33 \times 10^{-14} I\lambda^2(\mathrm{eV}), \tag{4}$$

where U_{p} is the ponderomotive potential of radiation field and refers to the quiver motion of electron in an oscillating field, I is the laser intensity in $\mathrm{W/cm^2}$, λ is the laser wavelength in μm. The electron-ion/neutral collision frequency inside a cluster is on the order of 10^{14} due to near-liquid density ($10^{21} - 10^{22}/\mathrm{cm^3}$). From the above analysis, the heating rate of the electron can reach more than 1000 eV $\mathrm{ns^{-1}}$ when the intensity of the laser is about $4.8\times10^{11}\,\mathrm{W/cm^2}$.

The ionization potentials (IPs) of S^{q+} (q = 1–6) are 10.36, 23.33, 34.83, 47.3, 72.68 and 88.05 eV, respectively. The IPs of C^{q+} (q = 1–4) are 11.26, 24.38, 47.89 and 64.49 eV, respectively. So it takes only hundreds of picoseconds to heat an electron up to a kinetic energy that exceeds the ionization potential of stepwise ionization process (less than 100 eV). When the electrons gain enough energy to exceed the corresponding IP, electron-impact ionization (EI) can take place. Some of the electrons with high kinetic energies may also escape from the cluster, leaving the highly charged cluster ions. These heating and ionization processes circulate. When the repulsive energy of like charges overcomes the total cohesive energy of cluster, the Coulomb explosion occurs, generating the multiply charged atomic ions. Some of the escaping electrons can also impact on, thus ionize the surrounding particles to generate some singly- or multiply- charged ions.

Our experimental results and the reason why no multicharged ions were observed in other MPI studies[1,5] can be easily understood using the above mechanism. First, the cluster is essential to the generation of multiply charged atomic ions, and the intensity of multicharged ions increases with increasing the concentration and size of clusters in the beam, when the backing pressure of carrier gas is increased or the laser is adjusted so that it irradiates the densest part of the pulsed molecular beam. Similar phenomena were also observed in the photoionization of seeded methyl iodide cluster beam by an intense 795 nm femtosecond laser with intensities varying from 2×10^{14} to $8\times10^{14}\,\mathrm{W/cm^2}$ by Ford *et al.*[22] Second, the angular isotropic distribution of the multicharged ions is easy to understand, as the electron-recollision-induced impact ionization is a dominant mechanism for the production of multicharged ions, which is independent of the polarization of the laser. So, this is different from the field ionization, i.e. barrier suppression or tunnelling ionization.

The above mechanism can explain why there are no multicharged ions observed when nanosecond laser, separately, at wavelengths of 193, 266 and 345 nm irradiates a CS_2 cluster beam. The heating rate of free electron in a cluster is scaled with λ^2, where λ is the wavelength of laser. The heating rates in the experiments of Patsilinakou *et al*[1] and Seaver *et al*[5] were about two orders smaller than those of our experiment, as the wavelength in is about 3–4 times larger and the laser intensities are also about ten times stronger. The electrons in the cluster can only be heated to several eV after the MPI in their experiments, which is smaller than the ionization potentials of parent ion and the singly charged atomic ions. The longer laser wavelength benefits the energy gaining of the electrons in the cluster, and increase the impact ionization rate and final ionization level.

In summary, the following processes account for the production of different ion species:

$$(\mathrm{CS_2})_n \xrightarrow{\mathrm{MPI}} (\mathrm{CS_2})_n^+ + \mathrm{e}, \tag{5}$$

$$(\mathrm{CS_2})_n^+ \xrightarrow{\mathrm{MPID}} \mathrm{CS_2^+} + \mathrm{S^+} + \mathrm{CS^+} + \mathrm{C^+}, \tag{6}$$

$$(\mathrm{CS_2})_n^+ + \mathrm{e} \xrightarrow{\mathrm{IB-EI}} (\mathrm{CS_2})_m^{z+} \xrightarrow{\mathrm{CE}} \mathrm{C}^{p+} + \mathrm{S}^{q+}, \quad (p = 2-4; q = 3-6), \tag{7}$$

$$\mathrm{CS_2} + \mathrm{e(escaping\ from\ the\ cluster)} \xrightarrow{\mathrm{EI}} \mathrm{CS_2^+} + \mathrm{CS^+} + \mathrm{S^+} + \mathrm{CS_2^{2+}} + \mathrm{S^{2+}}, \tag{8}$$

$$\mathrm{He} + \mathrm{e(escaping\ from\ the\ cluster)} \xrightarrow{\mathrm{EI}} \mathrm{He^+} + 2\mathrm{e}. \tag{9}$$

The ionization of cluster is ignited by multiphoton ionization as indicated in reaction (5). Reaction (6) can contribute to ion fragmentation. In our investigations, reaction (7) probably takes place after ionization. The escaping electrons can collisionally ionize the surrounding CS_2 molecules and He atoms to produce CS_2^+, CS^+, S^+, S^{2+} and He^+ with almost no kinetic energy when these electrons have enough energies, as shown in processes (8) and (9).

5. Conclusions

The formation of multicharged C^{q+} and S^{q+} from CS_2 clusters irradiated by a nanosecond laser field has been studied in detail by time-of-flight mass spectrometer. Multiphoton ionization (MPI) triggering - inverse bremsstrahlung (IB) heating - electron impact (EI) ionization has been proposed to explain the generation of these multicharged ions at the present laser intensity. The observation of multicharged-ions-ionized a CS_2 cluster beam under a nanosecond laser at 1064 nm shows that the longer wavelength of laser benefits the energy gaining of the electrons in clusters, as the heating rate of a electron in cluster, i.e. absorption energy from the laser field via IB, is proportion to λ^2, the square of laser wavelength.

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