

# Selective IR multiphoton dissociation of CF<sub>3</sub>I in a nonequilibrium pressure shock

G N Makarov, S A Mochalov, A N Petin

**Abstract.** The isotopically selective IR multiphoton dissociation of CF<sub>3</sub>I is studied in a nonequilibrium pressure shock formed in the interaction of a pulsed gas-dynamically cooled molecular flow with a solid surface. The yield of a C<sub>2</sub>F<sub>6</sub> product and its enrichment factor by the <sup>13</sup>C isotope were measured experimentally. The time-of-flight spectra of CF<sub>3</sub>I were obtained in the flow interacting with the surface and in an unperturbed flow. The spectral dependences of the yield of a C<sub>2</sub>F<sub>6</sub> product in an unperturbed flow and in a pressure shock were studied along with the process selectivity. It was shown that the efficiency of isotopically selective molecule dissociation can be significantly increased due to the formation of a pressure shock. The origin of the results observed are discussed.

**Keywords:** isotopically selective IR dissociation of molecules, gas-dynamically cooled molecular flow, interaction of a flow with a surface.

## 1. Introduction

It is known that cooling molecules in gas dynamic jets and flows [1] can substantially improve the selectivity of IR multiphoton excitation and dissociation [2–4]. However, the efficiency of photochemical processes in jets and flows is low. Owing to a low molecular concentration and a low gas temperature, the rates of chemical reactions are low, including those responsible for the formation of target products. In specific cases at a low concentration of molecules in the flow, a substantial fraction of radicals are lost at the walls without the formation of products (for instance, upon the dissociation of CF<sub>3</sub>I [5–7]).

A more favourable situation may be realised when a flow interacts with a solid surface. Upon the interaction of a flow with a surface, a pressure shock (a shock wave) is formed in front of the surface [8–10]. The nonequilibrium conditions in the pressure shock may be the reverse of the nonequilibrium conditions in the incident (unperturbed) flow

( $T_{1tr} \leq T_{1rot} \leq T_{1vib}$  in the incident flow and  $T_{2tr} \geq T_{2rot} \geq T_{2vib}$  in the pressure shock;  $T_{itr}$ ,  $T_{irot}$  and  $T_{ivib}$  are the translational, rotational, and vibrational molecular temperatures, respectively). The formation of the pressure shock in front of the surface results in a significant increase in the dissociation yield of molecules excited in the incident beam. In the pressure shock itself, new nonequilibrium conditions are realised which are of interest in studies of selective photochemical processes.

As shown recently [11], in the case of excitation of molecules in the flow incident on a surface, the product yield increases substantially (several-fold) owing to the formation of a pressure shock (compared to excitation of molecules in an unperturbed flow), while the process selectivity remains almost invariable. In Refs [12, 13], the selective dissociation of SF<sub>6</sub> in a pressure shock was studied for the first time. The yield of SF<sub>4</sub> was found to increase significantly (by more than an order of magnitude) compared to the case of excitation of molecules in an unperturbed flow, a decrease in the process selectivity being small (by 25–30%).

A CF<sub>3</sub>I molecule is, along with SF<sub>6</sub>, has been most studied from the viewpoint of IR multiphoton excitation and dissociation. The isotopically selective dissociation of CF<sub>3</sub>I has been also studied in a pulsed gas-dynamically cooled molecular flow [5–7, 14]. In this connection of interest is the investigation of selective CF<sub>3</sub>I dissociation under the nonequilibrium conditions of a pulsed flow interacting with a solid surface, which is the concern of our work.

## 2. Nonequilibrium conditions in a pressure shock

The ‘effective temperatures’ in gas-dynamically cooled molecular jets and flows typically satisfy the condition [1]

$$T_{1tr} \leq T_{1rot} \leq T_{1vib}. \quad (1)$$

In a pressure shock [8–10] formed upon the interaction of a pulsed flow with a surface, because of a difference between translational, rotational, and vibrational relaxation rates [15], the nonequilibrium conditions opposite to relation (1) may be realised:

$$T_{2tr} \geq T_{2rot} \geq T_{2vib}. \quad (2)$$

When a pulsed rarefied gas flow is used, the vibrational temperature of molecules in a pressure shock may virtually coincide with their vibrational temperature in the incident flow ( $T_{2vib} \approx T_{1vib}$ ) owing to a long vibrational-translational

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relaxation time (for instance, for  $\text{CF}_3\text{I}$   $p\tau_{\text{VT}} \approx 350 \pm 100$   $\mu\text{s Torr}$  [16]), whereas  $T_{2\text{tr}} > T_{1\text{tr}}$  and  $T_{2\text{rot}} > T_{1\text{rot}}$ . Therefore, new nonequilibrium conditions are produced in a pressure shock under which the vibrational temperature of molecules is significantly lower than their translational and vibrational temperatures. It was under these conditions that the selective  $\text{CF}_3\text{I}$  dissociation was studied in our work.

### 3. Experimental

The experimental setup and method are described in detail in Refs [5–7, 12]. Here, we outline only the main points. A pulsed nozzle of the ‘current loop’ type [17] was employed to obtain the flow of molecules. The nozzle orifice was 0.75 mm in diameter and the nozzle gating pulse was 100  $\mu\text{s}$  in duration (FWHM). The gas pressure above the nozzle could be varied in the 0.1–3.5 bar range. The number  $N_f$  of molecules exhausting from the nozzle per pulse depended on the gas pressure above the nozzle and was varied from  $\sim 5 \times 10^{15}$  to  $1.2 \times 10^{17}$  per pulse in these experiments. The nozzle could be operated either in a single-pulse mode or with a repetition rate up to 1 Hz.

The vacuum chamber (of volume  $V_{\text{ch}} \approx 20$  litre), in which the flow of molecules was formed, was evacuated to a pressure of  $(1-2) \times 10^{-6}$  Torr by a diffusion pump with a pumping rate of 500 litre  $\text{s}^{-1}$ . The flow of molecules was formed with the aid of two thin metal strips attached to the outlet nozzle cone in such a way that they formed a dihedral angle with the edge parallel to the  $y$ -axis. They possessed a varied radius of curvature in the  $xz$  plane. The average velocity  $v_x$  of the flow of  $\text{CF}_3\text{I}$  molecules in the excitation region (at a distance  $x \geq 50$  mm from the nozzle) was  $400 \pm 20$   $\text{m s}^{-1}$ .

The solid surface (we used KBr and  $\text{CaF}_2$  crystal plates) was placed at a distance of  $\sim 50-150$  mm from the nozzle, perpendicular to the flow direction. The interaction of the supersonic flow with the surface resulted in the formation of a pressure shock (a shock wave) in front of it [8–10], the condition in the shock being essentially nonuniform, non-stationary, and nonequilibrium. The characteristic dimension of the pressure shock front, by the order of magnitude equal to the molecule mean free path [8, 9], was 0.2–5 mm under our experimental conditions.

Molecules were excited by a tunable TEA  $\text{CO}_2$  laser. The laser pulse energy was 3 J. The molecules were excited near the surface, at a distance  $\Delta x = 1.5-8$  mm from it. The laser radiation was focused into this region by a cylindrical lens with a focal length of 25 cm. The lens axis was parallel to the surface, the cross section area of the laser beam at the lens focus measured  $\sim 0.24$   $\text{mm} \times 12.5$  mm.

We measured the  $\text{C}_2\text{F}_6$  yield and its  $^{13}\text{C}$  isotope enrichment factor. The procedure of collection, IR and mass analysis of the products and of the gas remaining after irradiation and the molecule dissociation in the gas-dynamic flow is described in detail in Refs [5–7]. The isotope composition of  $\text{C}_2\text{F}_6$  was determined from  $\text{C}_2\text{F}_5^+$  ion fragments and the enrichment factor was defined as

$$K_{13}^{\text{prod}} = \frac{2I_{121} + I_{120}}{(I_{120} + 2I_{119})\zeta}, \quad (3)$$

where  $I_{119}$ ,  $I_{120}$  and  $I_{121}$  are the intensities of the  $\text{C}_2\text{F}_5^+$  ion-mass peaks; and  $\zeta \approx 0.011$  is the carbon isotope  $^{13}\text{C}$  and  $^{12}\text{C}$  contents ratio in the initial  $\text{CF}_3\text{I}$  gas.

## 4. Results and discussion

### 4.1. Measurements of the flow parameters

The main parameters that influence the formation of the pressure shock in front of the surface are the velocity  $v_x$  of the molecule flow, its extent  $\Delta x_f$ , and the flow molecule density (concentration)  $N_1$ . The average flow velocity and extent were measured by the time-of-flight technique [18, 19] using a pyroelectric detector. In addition, they were determined from the dependence of the  $\text{C}_2\text{F}_6$  yield on the time delay  $\tau_d$  between the nozzle gating pulse and the exciting laser pulse in the case of  $\text{CF}_3\text{I}$  excitation in an unperturbed flow.

The concentration  $N_1$  of molecules in the flow was estimated from the measured total number  $N_f$  of particles issuing from the nozzle in one pulse and the calculated flow volume  $V_f$  ( $N_1 \approx N_f/V_f$ ). The method of determining the number  $N_f$  of molecules is described in detail in Refs [5, 7]. The flow volume  $V_f = S\Delta x_f$  was estimated from its geometry ( $S$  is the flow cross section). The cross section flow area at  $\sim 50$  mm from the nozzle was 7.2  $\text{cm}^2$  and the flow extent was  $\sim 4$  cm. Hence,  $V_f \approx 30$   $\text{cm}^3$ . The parameters of the  $\text{CF}_3\text{I}$  molecule flow at a distance  $x \approx 50$  mm from the nozzle are given below.

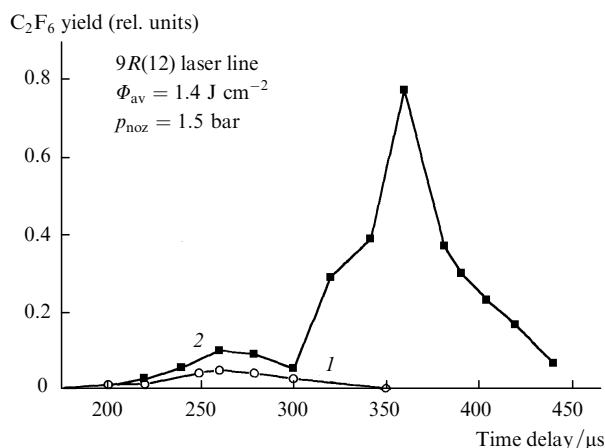
Pulse duration $\Delta\tau_f$ .....	$\sim 100$ $\mu\text{s}$
Flow extent $\Delta x_f$ .....	$\sim 4$ cm
Velocity $v_x$ .....	$400 \pm 20$ $\text{m s}^{-1}$
Number of molecules $N_f$ .....	$5 \times 10^{15} - 8 \times 10^{16}$
Volume $V_f$ .....	$\sim 30$ $\text{cm}^3$
Molecule density $N_1$ .....	$1.7 \times 10^{14} - 2.7 \times 10^{15}$ $\text{cm}^{-3}$

### 4.2. Dissociation of $\text{CF}_3\text{I}$ in the flow interacting with a surface

Fig. 1 shows the  $\text{C}_2\text{F}_6$  yield as a function of the time delay  $\tau_d$  between the nozzle gating pulse and the laser radiation pulse upon  $\text{CF}_3\text{I}$  excitation in an unperturbed flow (curve 1) and in the flow interacting with a surface (curve 2). The molecules were excited at a frequency of 1073.3  $\text{cm}^{-1}$  [the 9R(12) laser line], at resonance with the  $\nu_1$  molecular vibration [20]. The gas pressure above the nozzle was 1.5 bar. The distance from the nozzle to the surface was 51 mm and  $\Delta x = 2.5$  mm.

One can see that when  $\text{CF}_3\text{I}$  is excited in the flow interacting with the surface, the  $\text{C}_2\text{F}_6$  yield is higher than upon excitation of molecules in the unperturbed flow for all time delays  $\tau_d$ . For short time delays ( $\tau_d \leq 300$   $\mu\text{s}$ ), when the pressure shock did not reach yet the excitation region, the increase in the  $\text{C}_2\text{F}_6$  yield is caused by the increase in the  $\text{CF}_3\text{I}$  dissociation yield arising from the collisions between excited molecules in the pressure shock [11, 13].

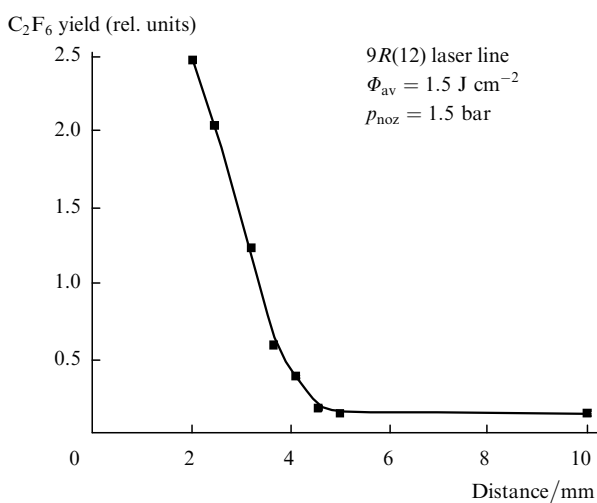
Upon the IR multiphoton excitation, an ensemble of highly excited molecules is formed, which has a relatively broad distribution over the excited states [2–4]. As a result, a part of the molecules dissociates radiatively and a part dissociates owing to collisions of excited molecules with one another. In an unperturbed flow, only a radiative dissociation occurs, while a collision dissociation of highly excited molecules, whose contribution to the total yield is usually very large (see, e.g., Ref. [2]), does not occur due to the deficit of collisions.



**Figure 1.** Dependence of the C<sub>2</sub>F<sub>6</sub> yield on the time delay  $\tau_d$  between the nozzle gating pulse and the laser radiation pulse upon excitation of CF<sub>3</sub>I in the unperturbed flow (1) and in the flow interacting with the surface (2).

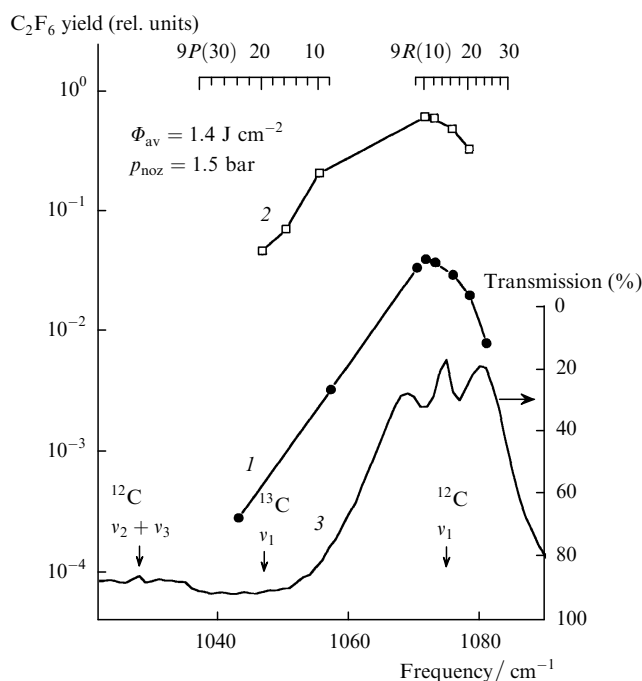
The molecules excited in the incident flow find themselves in the pressure shock, where the particle density and the gas temperature are significantly higher than in an unperturbed flow. This is the reason why the conditions for collisions between highly excited molecules are realised in the pressure shock, resulting in a higher dissociation yield. When molecules are excited in the pressure shock itself (for  $\tau_d \approx 325 - 400 \mu$ s), the C<sub>2</sub>F<sub>6</sub> yield is substantially higher than in the case of their excitation in the unperturbed flow. In particular, the C<sub>2</sub>F<sub>6</sub> yield at the peak of the dependence represented by curve 2 (for  $\tau_d = 360 \mu$ s) is approximately 15 times higher than in the unperturbed flow.

As the distance  $\Delta x$  from the excitation region to the surface was shortened (lengthened), the C<sub>2</sub>F<sub>6</sub> yield in the pressure shock increased (decreased) and the delay time  $\tau_d$  for which the C<sub>2</sub>F<sub>6</sub> yield peaked shortened (lengthened). Fig. 2 shows the dependence of the C<sub>2</sub>F<sub>6</sub> yield on the distance  $\Delta x$ . It characterises the width and the steepness of the pressure shock. One can see that the front width is  $\sim 3.5 - 4 \text{ mm}$  for a CF<sub>3</sub>I pressure of 1.5 bar above the nozzle and a nozzle–surface distance of 51 mm.



**Figure 2.** Dependence of the C<sub>2</sub>F<sub>6</sub> yield on the distance  $\Delta x$  upon excitation of CF<sub>3</sub>I in the pressure shock.

Fig. 3 shows the spectral dependences of the C<sub>2</sub>F<sub>6</sub> yield in the cases when CF<sub>3</sub>I is excited in an unperturbed flow (curve 1) and in the pressure shock (curve 2). For all the frequencies involved, the C<sub>2</sub>F<sub>6</sub> yield is seen to be significantly higher in the case when CF<sub>3</sub>I is excited in the pressure shock than in the case of excitation of the molecules in the unperturbed flow. In the latter case, the peak C<sub>2</sub>F<sub>6</sub> yield [at the 9R(10) and 9R(12) lines] is 12–15 times higher; in the low-frequency wing [for instance, at the 9P(20) line], which coincides with the <sup>13</sup>CF<sub>3</sub>I absorption band, the yield is higher by more than a factor of 200. So enormous a difference of the C<sub>2</sub>F<sub>6</sub> yields in the excitation of molecules in the distant wing of the spectrum is related primarily to a strong dependence of the product yield on the density of molecules under irradiation [5, 21] resulting from the C<sub>2</sub>F<sub>6</sub> production in binary collisions of the CF<sub>3</sub> radicals. Another reason why the difference is so great lies with a relatively high rotational CF<sub>3</sub>I temperature in the pressure shock compared to that in the unperturbed flow (see the estimates below).



**Figure 3.** Dependences of the C<sub>2</sub>F<sub>6</sub> yield on the laser radiation frequency upon excitation of CF<sub>3</sub>I in an unperturbed flow (1) and in the pressure shock (2) for a nozzle–surface distance of 51 mm,  $\Delta x = 2.5 \text{ mm}$ . The CF<sub>3</sub>I linear-absorption spectrum (3) the positions of CO<sub>2</sub> laser lines (at the top) are also shown.

The process selectivity was investigated by measuring the <sup>13</sup>C isotope enrichment factor of C<sub>2</sub>F<sub>6</sub> upon excitation of CF<sub>3</sub>I in the flow incident on the surface, in the pressure shock, and also in the unperturbed flow. The molecules were excited at a frequency of  $1046.85 \text{ cm}^{-1}$  [the 9P(20) CO<sub>2</sub> laser line] at resonance with the  $\nu_1$  vibration of <sup>13</sup>CF<sub>3</sub>I [20]. It was at the 9P(20)–9P(24) lines that we observed the highest enrichment factor in C<sub>2</sub>F<sub>6</sub> earlier [5]. The results obtained, are collected in Table 1 along with the product yield data. When the molecules were excited in the unperturbed flow, the enrichment factor  $K_{13}^{\text{prod}}$  was  $21 \pm 3$  for an energy density of  $\sim 1.5 \text{ J cm}^{-2}$ . When the excitation was accomplished in the pressure shock,  $K_{13}^{\text{prod}} = 15 \pm 3$ .

**Table 1.** Yield of the C<sub>2</sub>F<sub>6</sub> product and its <sup>13</sup>C isotope enrichment factor upon dissociation of CF<sub>3</sub>I in an unperturbed flow and in the flow interacting with a surface (the CF<sub>3</sub>I pressure above the nozzle is 1.5 bar and the energy density 1.5 J cm<sup>-2</sup>).

CO <sub>2</sub> laser line	C <sub>2</sub> F <sub>6</sub> yield (rel. units)			Enrichment factor for C <sub>2</sub> F <sub>6</sub> , K <sub>13</sub> <sup>prod</sup>		
	Unperturbed flow	Incident flow	Pressure shock	Unperturbed flow	Incident flow	Pressure shock
9R(12)	1 ± 0.2	2.5 ± 0.5	14 ± 3	–	–	–
9P(20)	–	–	–	21 ± 3	19 ± 3	15 ± 3

### 4.3. Discussion of results. Evaluation of the density and the temperature of CF<sub>3</sub>I in the pressure shock

We emphasise that the strongly nonuniform, nonstationary, and nonequilibrium conditions in the pressure shock and the numerous processes occurring therein make it difficult to interpret the results obtained. That is why we will restrict ourselves to their qualitative discussion and perform crude estimates of the density and the effective temperature of gas in the pressure shock.

The increase in product yields with the excitation of molecules in the pressure shock may result from an increase in the gas density, the chemical reaction rate, and also the yield of dissociation of the molecules. The latter is due to the higher efficiency of their excitation in the pressure shock and to the collisional dissociation of the molecules excited by an IR pulse below the dissociation limit, which do not dissociate in the unperturbed flow owing to the deficit of collisions. The relatively high selectivity in the pressure shock results from the fact that the vibrational temperature of the molecules in it remains relatively low.

For a gas with a constant heat capacity, the limiting density increase in a normal shock is defined by the relationship [8–10]  $\rho_2/\rho_1 = (\gamma + 1)/(\gamma - 1)$ , where  $\rho_1$  and  $\rho_2$  are the gas densities in the incident flow and in the pressure shock, respectively;  $\gamma = c_p/c_V$  is the ratio of specific heat capacities. For CF<sub>3</sub>I at  $T \approx 300$  K,  $\gamma \approx 1.13$  [22, 23], and hence it follows  $\rho_2/\rho_1 \approx 17$ . In our case, however, not all degrees of freedom of a molecule contribute to the heat capacity, and therefore the ratio  $\rho_2/\rho_1$  is supposedly well below the above value.

We shall estimate the density of the pressure shock and the average density of molecules starting from the molecular flow parameters obtained in Section 4.1. For a crude estimate, the  $\rho_2/\rho_1$  may be assumed to be equal to the ratio between the flow extent ( $\sim 4$  cm in the excitation region, at a distance of 51 mm from the nozzle) and the width of the shock front ( $\sim 3.5$  mm, see Fig. 2), and so  $\rho_2/\rho_1 \approx 11$ . For a CF<sub>3</sub>I pressure of 1.5 bar above the nozzle, the total number of molecules in the flow is  $N_f \approx 5.2 \times 10^{16}$  and their density is  $N_1 \approx 1.7 \times 10^{15}$  cm<sup>-3</sup>. Hence, the average molecule density in the pressure shock is  $N_2 \approx 1.9 \times 10^{16}$  cm<sup>-3</sup>. The majority of experiments were performed for a distance  $\Delta x = 2.5$  mm to the surface. We note that the limiting density was not yet reached in this case (for shorter  $\Delta x$ , the C<sub>2</sub>F<sub>6</sub> yield was higher, see Fig. 2). This implies that the increase of the C<sub>2</sub>F<sub>6</sub> yield in the pressure shock is supposedly related not only to the increase of gas density, but also to other factors considered above.

The increase in gas temperature in the pressure shock arising from deceleration may be estimated [10] as  $\Delta T = v_0^2/2c_p$ , where  $v_0$  is the flow velocity and  $c_p$  is the heat capacity of a gas. We substitute the corresponding values for CF<sub>3</sub>I ( $v_0 \approx 400$  m s<sup>-1</sup>,  $c_p \approx 335$  J kg<sup>-1</sup> K<sup>-1</sup> [22, 23]) to obtain  $\Delta T \approx 240$  K. However, the increase in gas temperature is likely to exceed the resultant value because the heat

capacity of CF<sub>3</sub>I in the gas-dynamically cooled flow is below the value specified for  $T \approx 300$  K. From the energy conservation law for the CF<sub>3</sub>I molecules in the incident flow and in the pressure shock (under the assumption that the vibrational degrees of freedom have no time to heat up) it follows that the increase in temperature of the translational and rotational degrees of freedom is  $\Delta T \approx 580$  K. So, if the translational and rotational temperatures in the incident flow are  $T_{1tr} \approx T_{1rot} \approx 40$  K, the temperatures in the pressure shock are  $T_{2tr} \approx T_{2rot} \approx 620$  K. It seems likely that the vibrational molecule temperature  $T_{1vib}$  in the pressure shock is, as noted in Section 2, hardly different from that in the incident flow, where it was supposedly no higher than 150 K.

Consequently, if the vibrational molecule temperature is the dominant factor in the formation of selectivity, the selectivity of dissociation in the pressure shock may not differ strongly from that in the incident flow, which is what we observed in our experiments. Also note that the lowering of selectivity in the pressure shock owing to the increase in gas temperature may in specific cases be compensated for by its growth arising from the increase of density of the molecules under irradiation [21].

## 5. Conclusions

Therefore, the efficiency of selective IR multiphoton dissociation CF<sub>3</sub>I in a pulsed gas dynamic flow can be significantly improved through the formation of a pressure shock in the interaction of a flow with a solid surface.

The technique of pressure shock formation outlined opens up the opportunity to study selective photochemical processes in nonequilibrium conditions, which are opposite to the nonequilibrium conditions in gas-dynamically cooled jets and flows, namely, for  $T_{tr} \geq T_{rot} \geq T_{vib}$ .

The technique proposed for improving the efficiency of multiphoton dissociation appears to be advantageous in the selective dissociation of large polyatomic molecules (e.g., (CF<sub>3</sub>)<sub>3</sub>CX, where X is a halogen or hydrogen), which have rather long lifetimes of over 100–200  $\mu$ s relative to a monomolecular dissociation even for vibrational excitation energies significantly higher than the dissociation energy [24, 25]. For long lifetimes relative to decay, overexcited molecules manage to fly up to the chamber walls and relax on them without the formation of products. By forming a pressure shock, it is possible to realise the collisional dissociation of molecules and thereby rise the product yields.

To summarise, we note that the vibrational–translational relaxation in some polyatomic molecules occurs rather fast, which imposes limitations on the application of the method outlined. As a consequence, a fast heating of also the vibrational molecular degrees of freedom will take place in the pressure shock. This will result in a lowering of the dissociation selectivity if the molecules are excited in the

pressure shock itself. However, in the case when the molecules are excited in the flow incident on the surface, the process efficiency can nevertheless be improved (compared to the case of excitation of the molecules in an unperturbed flow) due to the collisional dissociation of highly excited molecules in the pressure shock forming in front of the surface.

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