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Generation of singlet oxygen in fullerene-containing media: 1. Photodesorption of singlet oxygen from fullerene-containing surfaces^{*}

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Abstract. It is shown that upon irradiation of fullerenecontaining surfaces by laser or flashlamp pulses, oxygen adsorbed by these surfaces efficiently escapes to the gas phase. The observation of luminescence pulses in the spectral region of 762 and 1268 nm confirms the presence of oxygen molecules in the excited singlet state in the desorbed oxygen. The conditions for optimisation of the efficiency of singletoxygen production are studied. It is shown that singlet oxygen at the concentration sufficient for obtaining operation of a fullerene-oxygen-iodine laser can be produced in this way.

Keywords: fullerene, singlet oxygen, photodesorption, luminescence.

1. Introduction

The problem of studying photoinduced processes at the gas-solid interface was posed by A.N. Terenin as early as the 1920s for applications in photochemistry, in particular, photosynthesis and catalysis. The recent development of nanotechnologies has stimulated a renewed interest in these processes. The interaction of the atmospheric oxygen with various surfaces used as photosensitisers occupies an important place in this field.

The photoinduced generation of singlet oxygen by the deposited layers of sensitisers, which can be various dyes [1, 2], fullerenes [3, 4], and fullerene-like materials (nano-tubes and astralenes – fullerene-like multilayer polyhedral particles of size ~ 100 nm) [5, 6] is now rather widely used to produce singlet oxygen, in particular, ejected to the gas phase. In addition, the photodesorption of oxygen is used for purification of various electronic devices based on fullerenes and nanotubes [7-9], where the sorption of the atmospheric oxygen plays the negative role.

The study of the oxygen photodesorption to the gas phase from the surface of fullerenes and fullerene-like

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particles (astralenes and nanotubes) used as singlet-oxygen sensitisers is of interest, in particular, for the development of an ecologically pure fullerene-oxygen-iodine laser [10-12]. In this paper, we continue investigations of the photodesorption of oxygen from the fullerene and astralene surfaces, which were started in paper [6], where the generation of singlet oxygen was studied by observing luminescence at wavelengths 762 and 1268 nm corresponding to the ${}^{1}\Sigma_{g}^{+} \rightarrow {}^{3}\Sigma_{g}^{-}$ and ${}^{1}\Delta_{g} \rightarrow {}^{3}\Sigma_{g}^{-}$ transitions in O_{2} by exciting fullerene- and astralene-containing coatings by a flashlamp, in particular, in a laser cell. In addition, excitation was performed by a pulsed neodymium laser in a specially constructed continuous-flow gas vacuum cell. It was shown that such coatings could be used to obtain singlet oxygen at concentrations sufficient for obtaining generation in an oxygen-iodine laser [12]. The time dependence of the luminescence intensity of singlet $({}^{1}\Delta_{\sigma})$ oxygen was characterised by two regions: the initial region corresponding to emission from the surface with the characteristic decay time $1-2 \ \mu s$ and the region corresponding to the emission of singlet oxygen in the gas phase with the decay time $\sim 100 - 500 \ \mu s$ and longer. It was assumed that the singlet-oxygen desorption is governed by the thermal mechanism.

In this paper, we studied the photodesorption and luminescence of singlet oxygen in more details by investigating the dependences of desorption rate and luminescence intensity on the temperature, pressure and the gas type. In addition, we studied the luminescence spectra of singlet oxygen and the influence of the substrate material, coating and the type of adhesion of coating particles with a substrate on these processes.

2. Experimental

The influence of irradiation on the systems formed upon desorption of gas molecules adsorbed on surfaces is studied by various methods such as IR absorption spectroscopy, resonance multiphoton ionisation and photoelectronic UV spectroscopy [13]. In addition, semiconductor sensors of singlet oxygen are used [1, 2, 5, 7] and the luminescence spectra of singlet oxygen [3, 4, 6] and the phosphorescence spectra of substrates [14] are recorded. Additional information by using these methods can be obtained by varying the excitation wavelength.

In this paper, we used mainly the luminescence method. Photodesorption was studied by using several setups, which as a whole were similar to the experimental setup described in [15]. Samples were excited either by 12-µs pulses from a

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xenon flashlamp with the energy density over the entire emission spectrum of $1-3 \text{ J cm}^{-2}$ or by the 10-ns, 50-mJ second-harmonic pulses from a neodymium laser. The surface density of fullerenes on the irradiated surface upon flashlamp pumping was $0.35-3.5 \text{ mg cm}^{-2}$. Fullerene or astralene coatings were deposited on ground glass and porous metal or ceramic substrates. Luminescence was observed in the direction perpendicular to the pump laser beam and detected with a germanium photodiode. The luminescence lines of singlet oxygen at 762 and 1268 nm were selected by using interference filters or an MDR-4 monochromator with the spectral resolution of 5 nm (for the slit width of 1.2 mm). Luminescence was observed simultaneously from the surface itself and from gas leaving the surface, which resulted, as a rule, in a complicated temporal structure of luminescence pulses. The luminescence of oxygen escaped from the surface determined the longest time component of the signal, which could be always separated by analysing the time dependence of the luminescence intensity in a semi-logarithmic scale.

Coatings were prepared by using the powders of polycrystalline C_{60} , amorphous astralene, soot, and ground shungite (hard coal containing factions of percent of fullerene). The adsorbent powder was applied on a substrate by using a compound. As compounds, cements based on polystyrene and silicone were used. The coatings had a thickness of ~ 100 μ m and were opaque in the visible region.

3. Results and discussion

Before performing photodesorption experiments, we studied the dependences of the sorption ability of fullerene-containing coatings on temperature, gas pressure, and the coating preparation method [15-17].

Upon irradiation of adsorbents by intense laser or xenon flashlamp pulses, the characteristic emission of the irradiated surface was observed. The broadband emission spectrum exhibited the luminescence bands of singlet oxygen at 762 and 1268 nm along with the thermal emission of the surface heated by the pump pulse. In this case, singlet oxygen efficiently escaped to the gas phase, which was confirmed by the presence of the luminescence pulse of duration above 200 μ s. At the same time, the characteristic luminescence signal with the singlet-oxygen lifetime of $\sim 1-2 \ \mu$ s was observed at the initial (in time) interval of emission pulses.

Figure 1 shows the luminescence signals of singlet oxygen at 1268 nm observed upon its 530-nm laser-induced photodesorption from fullerene C_{60} -containing coatings deposited on various substrates. One can see that the luminescence intensity depends on the density of absorbing coatings deposited on substrates, and there is no considerable difference between these dependences for different substrates.

Figures 1a and b illustrate the process of photodesorption from dielectric substrates (porous ceramics and ground



Figure 1. Luminescence signals of singlet oxygen at 1268 nm observed upon its photodesorption from fullerene-containing coatings on porous ceramic substrates with the surface coating densities 3.5(1) and $0.5 \text{ mg cm}^{-2}(2)(a)$, ground glass substrates with the use of different compounds: polystyrene (1), 'liquid glass' (2), and silicone compound (3) (b), garnet substrates with the surface coating densities 3.5(1) and $2.5 \text{ mg cm}^{-2}(2)(c)$, a porous copper substrate with the use of the silicone compound (1) and without it (2) (d).

glass). Figures 1c and d present luminescence pulses observed in the case of graphite and porous copper substrates. Note that no noticeable difference between the luminescence pulses was observed for different porous metal substrates (Cu, Fe, Pb). Upon excitation of fullerene coatings on metals, the luminescence signals of singlet oxygen had smaller amplitudes and were shorter than in the case of ceramic and glass substrates, which is probably explained by a weaker heating of metal substrates due to their higher heat conduction, resulting in a weaker desorption of oxygen. The maximum duration of the luminescence signal was observed for glass substrates, the duration of the luminescence signal of singlet oxygen achieving 1 ms with increasing the pump intensity. The luminescence signal observed from the porous ceramic surface had the maximum amplitude simply because of the high density of the coating due to the type of the porous structure of the ceramics.

Compounds based on silicone or polystyrene were used to provide the higher coating density. However, it was found that compounds noticeably affected the parameters of luminescence pulses of singlet oxygen escaped to the gas phase, which is probably explained by the thermal decomposition of compounds and quenching of singlet oxygen by the decomposition products.

Figure 2 presents the luminescence signals of singlet oxygen observed upon irradiation of astralene samples. Unlike astralene samples studied earlier [15], which were deposited in the powder form on substrates, samples investigated here were prepared by pressing with the use of some binding materials. They represented plates of thickness 3-5 mm with any required transverse dimensions. The porous structure of these samples, obtained after their pressing, provided much stronger luminescence signals of singlet oxygen escaped to the gas phase compared to those observed earlier [15]. In addition, we note another feature observed in experiments with these samples, namely, the change in the luminescence intensity in a series of successive irradiations of samples by laser pulses. Such behaviour can be probably related to some specific features in the manufacturing technology of these samples.

To explain our results on the photodesorption of singlet oxygen from adsorbents, we calculated the heating temperature of the adsorbent surface irradiated by a light pulse.



Figure 2. Luminescence of singlet oxygen at 1268 nm for an astralene sample irradiated by a laser pulse train. Signals are obtained upon excitation by the first (1), third (2), fourth (3), and tenth (4) pump pulses.

The absorption coefficient of a solid fullerene-containing coating in the visible region is quite large $(10^3 - 10^5 \text{ cm}^{-1} \text{ [15]})$, and therefore we can calculate the temperature distribution inside the surface from the expression [18]

$$T(z,\tau) = \frac{\sqrt{\kappa}}{K\sqrt{\pi}} \int_0^\tau F(\tau-t) \exp\left(-\frac{z^2}{4\kappa t}\right) \frac{dt}{\sqrt{t}},$$

where $F(\tau - t)$ is the radiation flux density incident on the surface; *K* is the heat conductivity of the coating material; and $\kappa = K/(\rho c_p)$ is the thermal diffusivity. We assume in our estimates that the heat conductivity of fullerene is equal to the average value of the tabulated heat conductivities of graphite and soot [19]: $K = 1 \text{ J cm}^{-1} \text{ s}^{-1} \text{ K}^{-1}$. The density of crystalline fullerene $\rho = 1.7 \text{ g cm}^{-3}$ and its heat capacity $c_p = 0.5 \text{ J}^{-1} \text{ g}^{-1} \text{ K}^{-1}$ are well known [20].

The calculation performed with the function $F(\tau - t)$ simulating the laser or flashlamp laser pulse with the energy parameters presented above predicts the increase in temperature upon flashlamp pumping by 230 °C on the surface, by 230 °C at a depth of 10 µm, and by 30 °C at a depth of 100 µm. Therefore, the 10-µm-thick coating is heated more or less uniformly during the light pulse action. The penetration depth for visible radiation to a fullerenecontaining coating was approximately the same. The study of the temperature dependence of the sorption of oxygen by fullerene or astralene [17] (Fig. 3) shows that oxygen is strongly, if not completely, desorbed from the coating volume heated to 330-350 K. Thus, it is obvious that upon irradiation by light pulses, oxygen can escape to the gas phase not only from the coating surface but also from its volume at a depth of at least up to 50 µm. Based on this conclusion and the results of sorption measurements, we can obtain correct quantitative data on the escape of oxygen to the gas phase for any initial temperature of the fullerene or astralene coating. However, it is much more difficult to determine the fraction of singlet oxygen in this process taking into account that the thermal desorption of oxygen occurs not only from a depth of 10 µm reached by the exciting radiation, but also from the deeper layers of the sample heated due to heat conduction.

Note here that along with the usual thermal mechanism of gas desorption, oxygen in our case, because of the general increase in the adsorbent temperature upon pumping, the direct photodesorption of singlet oxygen can take place [5, 13] due to photoinduced electronic transitions in the adsorbate-substrate system. This is caused by the fact that



Figure 3. Isobars of the oxygen sorption on fullerene (1) and astralene (2) at the oxygen pressure 200 Torr.

during the transfer of excitation from fullerene to oxygen, the oxygen molecule can receive, simultaneously with the transition to the excited ${}^{1}\Sigma_{g}^{+}$ state, the additional kinetic energy, which can be sufficient for overcoming the binding energy between the adsorbate and substrate. In addition, the binding energy between the adsorbate and substrate can decrease itself upon the transition of gas to the excited state. However, in our case at high pump levels and strong adsorbent heating, the thermal desorption process dominates.

The estimates of the surface temperature performed for an adsorbent exposed to laser radiation show that the adsorbent surface irradiated by tightly focused laser beam can be heated up to very high temperatures (1500-3000 K), at which not only adsorbed gases can escape to the gas phase, but also partially the adsorbent material. Such effects were observed earlier, for example, in [21]. The emission of the heated adsorbent is distinctly observed in the luminescence spectra of $O_2(^{1}\Delta_g)$ in the region from 1150 to 1400 nm (Fig. 4). Figure 4 presents the luminescence spectra integrated over the observation time. The substrate is not damaged considerably in this case because during the short pump laser pulse only a layer of thickness a few micrometres has time to be heated. The amount of the evaporated material depends on the thermal properties of the coating and compound materials and also on the radiation energy density on the sample, which determines finally the intensity of local heating of the sample.

The dependence of the luminescence signals on the radiation energy density is illustrated in Fig. 5 for two different coatings. The total radiation energy was maintained constant, and only the irradiated surface area was varied. Figure 5 clearly demonstrates, first, the intense



Figure 4. Luminescence spectra of different fullerene-containing coatings: C_{60} on a glass plate with a 'liquid glass' compound for two radiation energy densities (a); C_{60} deposited from solution to a ceramic plate (1); C_{60} on a glass plate with polystyrene (2) and silicone (3) compounds for the radiation energy density 0.5 J cm⁻² (b).

luminescence signals of singlet oxygen from the adsorbent surface with the lifetime $\sim 1 - 2 \mu s$, second, the emission of the heated adsorbent material both from the surface and partially evaporated, whose duration (up to 100 μs) is determined by the rate of thermal relaxation inside the coating and in the volume of the evaporated material, and, third, the luminescence of singlet oxygen in the gas phase, the rate of oxygen escape to the gas phase being dependent on the pump energy density.



Figure 5. Luminescence signals from two coatings: C_{60} on a glass plate with a 'liquid glass' compound (a) and a polystyrene compound (b), for the radiation energy densities on samples 2.0 (1), 1.0 (2), 0.7 (3), and 0.5 J cm⁻² (4).

Note that the maximum escape of oxygen to the gas phase is realised for both coatings not at the maximum radiation energy density on the sample but at some intermediate value achieved by gradually increasing the irradiated area. This is probably explained by the fact that the surface heating required for the efficient escape of the adsorbed oxygen to the gas phase corresponds to the radiation energy density $0.7-1.0 \text{ J cm}^{-2}$ because the further increase in the irradiated area, as is shown in Fig. 5a, reduces the oxygen escape to the gas phase. The optimal radiation energy density and the duration of the luminescence signal depend on the coating type.

The question of the duration of the luminescence signal of singlet oxygen escaped to the gas phase should be considered in more detail. It is well known [22] that the lifetimes of the initial excited states of magnetic dipole ${}^{1}\Sigma_{g}^{+} \rightarrow {}^{3}\Sigma_{g}^{-}$ and ${}^{1}\Delta_{g} \rightarrow {}^{3}\Sigma_{g}$ transitions in oxygen in the absence of quenching collisions are very large (7 s for ${}^{1}\Sigma_{g}^{+}$ and 45 min for ${}^{1}\Delta_{g}$). Therefore, the very short duration of luminescence signals compared to these lifetimes can be explained only by quenching collisions in the volume from

which luminescence is observed. It was shown earlier in the study of the luminescence of singlet oxygen in the C_{60} solution in CCl₄ [23] that upon intense irradiation of the solution, a quencher was formed in it, which represented a complex of fullerene oxides and reduced the luminescence lifetime down to 500 µs from the initial lifetime ~ 50 ms. The concentration of this quencher was extremely low because no changes in the initial concentration of the solution was observed, which demonstrates a very high quenching efficiency.

Based on these data, we can reasonably assume that a similar quencher can be also produced upon irradiation of a solid fullerene surface. Note that the formation of fullerene oxides upon irradiation of solid fullerene samples has been already observed in [24-26]. However, the authors of these papers have not pointed out the relation between the formation of fullerene oxides and the luminescence lifetime of singlet oxygen, whereas we observed such a relation in fullerene solutions [2]. This allows us to use these facts to explain the luminescence lifetime of singlet oxygen observed in experiments. In the light of conclusions made above, we consider again the dependence of the luminescence signals of singlet oxygen on the radiation energy density on the sample in Fig. 5, which demonstrates that the luminescence lifetime noticeably increases with decreasing the radiation energy density and, hence, with the decrease in the production of oxides. In this case, the luminescence pulse shape also changes.

The luminescence spectra observed in experiments have the band in the region of the ${}^{1}\Delta_{g} \rightarrow {}^{3}\Sigma_{g}^{-}$ transition in oxygen. The amount of the observed singlet oxygen can be judged from the integrated intensity of this band. Unfortunately, the considerable uncertainty of experimental parameters (the angular distribution of the luminescence intensity, radiation loss in a monochromator, etc.) prevent accurate calculations of the singlet oxygen concentration produced on the surface; however, the obtained results can be used for semi-quantitative estimates and comparative analysis of the type of photodesorption from various coatings. According to such estimates, the maximum amount of singlet oxygen escaped to the gas phase from the unit surface to the near-surface volume bordering the region irradiated by laser pulses can achieve 1.5×10^{18} cm^{-2} . This value can be also determined, along with the singlet oxygen desorption from the surface, by contributions from oxygen excited over the surface by gaseous fullerene evaporated from the surface irradiated by high-power laser pulses.

It is obvious that such intense irradiation of the surface cannot be used for reliable permanent operation of a singlet oxygen generator because the fullerene coating is exhausted. Upon irradiation of the surface by a xenon flashlamp in a laser cell [12], the surface is heated up to 230 °C, as shown above. Fullerene is not evaporated from the surface at such temperature; however, the amount of produced singlet oxygen in this case cannot exceed the amount of oxygen molecules adsorbed at room temperature $(1.5 \times 10^{17} \text{ cm}^{-2})$. By comparing the quantitative results on singlet-oxygen generation obtained in our paper with the results obtained in earlier studies [3, 4] and analysing the possible reasons for differences observed, note first of all that these differences are mainly explained by different pump power levels. We used pulsed pumping with the high peak power, while the authors of papers [3, 4] used low-power cw pumping at

which the efficient thermal desorption of singlet oxygen and its escape to the gas phase could not occur because the surface was only very weakly heated by the pump radiation.

The luminescence spectrum in a broad spectral range shown in Fig. 6 confirms the presence of the broadband radiation resembling the black-body radiation. The approximation of the experimental spectrum by Planck's law of radiation gives the brightness temperature ~ 2000 K, in good accordance with our calculation presented above. Thus, we have confirmed experimentally that the experimental spectrum is determined by luminescence of singlet oxygen and thermal radiation of the heated adsorbent.



Figure 6. Luminescence spectrum of C_{60} on glass (1) and thermal blackbody radiation at T = 1800 K (2).

Note in conclusion that the luminescence spectrum of soot does not have a band at 1270 nm, which suggests that singlet oxygen is not produced upon optical excitation of soot. The luminescence spectrum of shungite exhibits a weak increase in the intensity at 1268 nm compared to the background intensity, which can be related to a small amount of singlet oxygen produced upon irradiation.

4. Conclusions

The study of the photodesorption and luminescence of singlet oxygen upon optical excitation of fullerene- and astralene-containing coatings has demonstrated the escape of oxygen from the adsorbent surface to the gas phase. The calculation of the heating temperature of the surface and experimental results confirm the thermal photodesorption mechanism. Luminescence spectra demonstrate the generation of singlet oxygen by such coatings upon their irradiation and the escape of oxygen to the gas phase during thermal desorption. Our estimates have shown that the maximum amount of singlet oxygen escaped from the unit surface to the near-surface volume in the gas phase bordering the irradiated region amounts to 1.5×10^{18} cm⁻² upon pulsed laser irradiation and to $1.5\times10^{17}~\text{cm}^{-2}$ upon irradiation by a xenon flashlamp. The analysis of the luminescence spectra allows one to compare the desorption abilities of coatings of different types, to study the influence of the compound and substrate materials on the quenching of singlet oxygen and to investigate the ablation resistance of coatings excited by high-power laser pulses. Based on the measurements performed in [24], the possible mechanism of the restriction of the luminescence lifetime (the lifetime of singlet oxygen in the gas phase) has been considered, which is related to the production of quenchers in the form of fullerene oxide complexes.

The existence of the optimal pump energy density is demonstrated at which the maximum escape of singlet oxygen to the gas phase is achieved. A considerable increase in the sorption of oxygen in fullerene at low temperatures [15-17] is also observed upon photodesorption of singlet oxygen, which allows the production of much higher concentrations of $O_2(^{1}\Delta_g)$ in the gas phase.

The study of the sorption properties of fullerenes and the photodesorption of singlet oxygen upon irradiation of adsorbents clearly demonstrate the possibility of using the obtained results in many fields in biology, medicine and laser technologies where reliable sources of singlet oxygen are required. This has been experimentally confirmed in papers [27] (medicine) and [6] (fullerene – oxygen – iodine laser).

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