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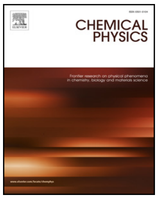

Luminescence of ND radicals during the destruction of molecular nitrogen nanoclusters

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ABSTRACT

In this work we studied optical spectra of thermoluminescence accompanying the destruction of collections of molecular nitrogen nanoclusters containing stabilized nitrogen, oxygen, and deuterium atoms. These collections of nanoclusters were formed by the injection of products of a radio-frequency discharge in deuterium-nitrogen-helium gas mixtures into bulk superfluid ^{4}He. In the range from 200 to 1650 nm, the bands at 336 nm, 473 nm along with a new band at 1170 nm as well as the known bands of atomic nitrogen and oxygen, and bands of molecular nitrogen, oxygen, and NO were observed. These three bands were assigned to the emission of the ND radicals formed due to recombinations of nitrogen atoms in excited metastable states and deuterium atoms in the ground state during the destruction of ensembles of molecular nitrogen nanoclusters.

1. Introduction

The method of matrix isolation provides possibilities for studying exotic highly unstable species. In the past, considerable efforts were made to create systems with high concentrations of matrix-isolated free radicals [1]. Different practical applications were proposed for those systems such as clean energy storage and more efficient rocket fuel. The best results for obtaining high concentrations of stabilized atoms were achieved by condensing gas-phase products of a radio-frequency discharge into bulk superfluid helium [2,3]. This method allows the formation of a porous structure consisting of a collection of impurity nanoclusters inside superfluid helium (HeII). The characteristic size of the impurity nanoclusters is of order 5 nm and the overall density of impurity atoms and molecules inside HeII is of order 10^{20} cm^{-3} [4–6]. The pores in the structures formed by ensembles of nanoclusters which are also known as impurity-helium condensates, have a broad distribution of sizes ranging from 8 to 860 nm [7–9]. All of the pores are filled with liquid helium. Usually, most of the stabilized atoms reside on the surfaces of the nanoclusters [10,11]. The highest concentrations of stabilized atoms were achieved in nitrogen–helium condensates with relative concentration of stabilized nitrogen atoms ($N/N_2 \times 100$ %) in the range 10–30% [12]. High concentrations of nitrogen atoms stabilized in molecular nitrogen nanoclusters provide substantial chemical energy for promoting recombination reactions of stabilized atoms during warming of the samples [13–15]. The rapid release of stored chemical energy resulted in rapid heating and intense thermoluminescence of the samples. In the past, studies of thermoluminescence during the destruction of samples were performed for impurity-helium condensates containing N_2 molecules, N atoms, and rare gas atoms, as well as small amounts of oxygen atoms. Strong emissions from N atoms (α, α', δ, δ'-groups), O atoms (β, β', β'' groups), N_2 molecules (VK bands), NO molecules (M and β-bands), XeO molecules (green bands), O_2 molecules (second Herzberg bands), and N^- anions (γ-line) were observed in these experiments [16–22]. These studies provided examples of chemical reactions at low temperatures in collections of nanoclusters containing stabilized nitrogen and oxygen atoms.

In recent experiments small admixtures of hydrogen or deuterium were added into gas mixtures used for preparation of nitrogen-helium condensates in superfluid helium [23]. A new intense broad band at $\lambda\sim$ 360 nm was observed in these experiments. This band was assigned to the $2A_g \rightarrow 1A_g$ transition of $N_4(D_{2H})$ polymeric nitrogen. Additionally, in the experiments with the introduction of deuterium, the bands at λ = 336 and 473 nm were observed and were tentatively assigned to the ($A^3\Pi_i \rightarrow X^3\Sigma^-$) and ($b^1\Sigma^+ \rightarrow X^3\Sigma^-$) transitions of the ND radical [23]. The transition ($A^3\Pi_i \rightarrow X^3\Sigma^-$) at λ = 471 had previously been observed in the emission spectra of the NH(ND) radicals in noble-gas matrices

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[24–27]. However in the same experiment we observed the γ-line emission at 793 nm, which corresponds to the $^1D_2 \rightarrow {}^3P_2$ transition of N^- anions [22]. The presence of N^- anions during the destruction of the samples raised the possibility of observing other transitions of this species. Therefore the transition $^1S_0 \rightarrow {}^3P_{1,2}$ of N^- anions may be an alternative interpretation for the line at λ = 473 nm. If we assign the line at λ = 473 nm to the N^- ($^1S_0 \rightarrow {}^3P_{1,2}$) transition, and knowing the wavelengths of another transition N^- anion ($^1D_2 \rightarrow {}^3P_{1,2}$) [22], it is possible to calculate the wavelength of the transition N^-($^1S_0 \rightarrow {}^1D_2$). The calculated wavelength for N^-($^1S_0 \rightarrow {}^1D_2$) transition is equal to λ = 1167 nm. The position of this line is very close to the position of the band corresponding to the transition $b^1\Sigma^+$ $v^{'} = 0 \rightarrow a^1\Delta^+$, $v^{''} = 0$ of the NH(ND) radical which was observed at λ = 1170.47 nm in an argon matrix [24].

To resolve the problem regarding the possibility of two alternative assignments of the emission at λ = 473 nm, we performed experiments with simultaneous registration of the spectra of emission in the ultraviolet (UV), visual and near infrared (NIR) regions during the destruction of pure nitrogen-helium samples and samples containing nitrogen and deuterium atoms. Analysis of these spectra provides convincing evidence for the assignment of the bands at λ = 336, 473, and 1170 nm to the ND radical.

2. Experimental setup

The experimental setup has been described in detail elsewhere [20]. Our experimental setup includes two concentric, silvered, glass Dewars. The inner Dewar is filled with liquid helium (LHe), and the outer Dewar with liquid nitrogen (LN_2). The helium Dewar is connected through a pumping line to an Edwards E2M80 mechanical pump. Using this pump, temperatures of 1.1 K are achievable inside the helium Dewar.

The gas mixtures that are used to create our samples are prepared at room temperature in a gas handling system. We increased the helium content of our gas mixtures to enhance the efficiency of dissociation of impurity molecules in the discharge. This system consists of a manifold connecting gas cylinders to gas mixing tanks, connection to an Edwards model 18 dual-stage rotary vane mechanical vacuum pump, and a Brooks 5850E mass flow controller. A stainless steel capillary connects the flow controller to the cryogenic portion of the setup. In our experiments we used Linde Electronics and Specialty Gases research-grade helium gas with 99.9999% purity. The oxygen present in our gas mixtures results from the ~1 ppm impurity in this gas.

Our samples are created by injecting a gas mixture into superfluid helium after passing through a 75 W radio-frequency (RF) ~50 MHz discharge zone. The RF discharge is provided by a HP 8656B signal generator supplemented with an Electronics & Innovations 3100L amplifier.

The discharge tube is made up of an outer quartz tube with an inner concentric quartz capillary. There are two discharge electrodes surrounding the capillary near the bottom of the quartz tube. The quartz tube is filled with LN_2 which simultaneously cools the discharge electrodes and the gas mixture passing through the capillary. The ~2 Torr pressure gradient between the discharge zone and the helium Dewar creates a well-formed jet as the excited atoms and molecules exit the orifice (ϕ~0.75 mm) of the discharge tube. Impurity atoms and molecules are rapidly cooled in the gas jet by the dense helium vapor and coalesce into nanoclusters. The beaker is placed ~2.5 cm below the orifice of the discharge tube. Inside the HeII, impurity nanoclusters aggregate into a porous structure. The level of HeII in the beaker is held constant by a thermo-mechanical fountain pump which supplies HeII from the bottom of the main bath. The temperature inside the sample beaker is maintained at 1.5 K by pumping on the main bath with the rotary pump, and is measured using a factory-calibrated Lake Shore Cryotronics GR-300-AA-0.3D germanium thermometer.

After the sample is created, the fountain pump is turned off, and the LHe is removed from the beaker by evaporation and the creeping film. When all of the LHe has left the beaker, the sample is said to be "dry". This process typically takes 15–20 min. With the dry sample, the valve to the pumping line is closed. Then the pressure and temperature increase inside the helium Dewar resulting in sample destruction, accompanied by luminescence of the sample, and concluding with bright flashes.

The emission of light in the UV and visible ranges is collected by a cryogenic fiber assembly [20] inside the Dewar which terminates at a vacuum feed-through. The opposite side of the vacuum feed-through is connected to a bifurcated fiber optical cable which simultaneously feeds an Ocean Optics HR2000 + spectrometer and an Andor SR500i spectrometer with a Newton EMCCD camera. The Ocean Optics spectrometer records spectra continuously in a wide spectral range (200–1100 nm) with a resolution of 1.3 nm. With this spectrometer the exposure time is generally between 250 and 500 ms. The Andor spectrometer can take high resolution spectra (0.53 nm, first grating) for a narrower spectral range ($\Delta\lambda$ = 340 nm), but has a much higher sensitivity, allowing for an exposure time of 3 ms during registration. We have not presented spectra obtained by the Andor spectrometer in this paper.

The spectra of NIR emission is observed from the outside of the Pyrex Dewars through a strip window in the silvering. A collection lens focuses light emitted by the sample onto the entrance of a collimating lens where a fiber optic cable is attached. The fiber connects to an Avantes NIR 512-1.7 TEC spectrometer. This spectrometer can record spectra continuously in a spectral range from 900 to 1650 nm, with an exposure time of 250–500 ms, and a resolution of 5 nm.

3. Experimental results

We studied spectra during the destruction of two samples prepared from gas mixtures: $[N_2]$:[He] = 1:100 and $[D_2]$:$[N_2]$:[He] = 1:2000:100000. Usually the process of sample destruction lasted ~65–70 s. Fig. 1 shows integrated spectra obtained during the destruction in the UV and visual spectral ranges. Addition of deuterium in nitrogen-helium gas mixtures leads to a substantial reduction in the intensities of all bands previously observed from nitrogen–helium samples (see Fig. 1). In the integrated spectra of the nitrogen-helium sample the α, α'-groups of N atoms, β, β', and β''-groups of O atoms, γ-line of N^- anions, Vegard-Kaplan (V-K) bands of N_2 molecules, M-bands of NO molecules and the second Herzberg bands of O_2 molecules are present [20]. Only a few bands such as the α, α'-groups of N atoms and the β-group of O atoms, and γ-line of N^- anions [22] remain in the

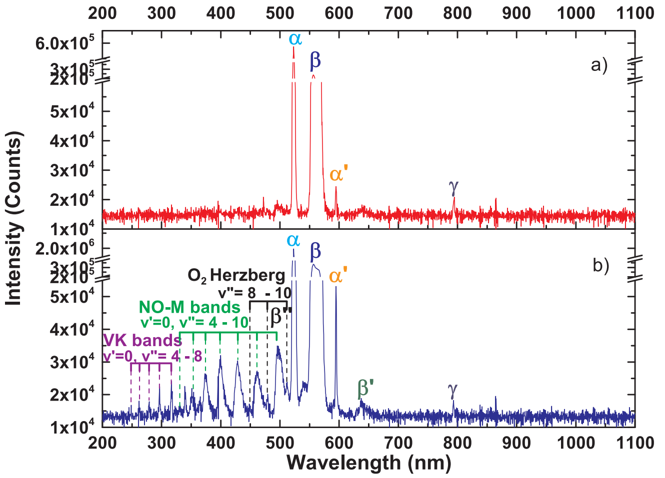

Fig. 1. Integrated spectra taken in the range 200–1100 nm by the Ocean Optics spectrometer during destruction of the samples prepared from different gas mixtures a) $[D_2]$:$[N_2]$:[He] 1:2000:100,000 (red), b) N_2:He 1:100 (blue). Each spectrum was accumulated during a period of 75 s.

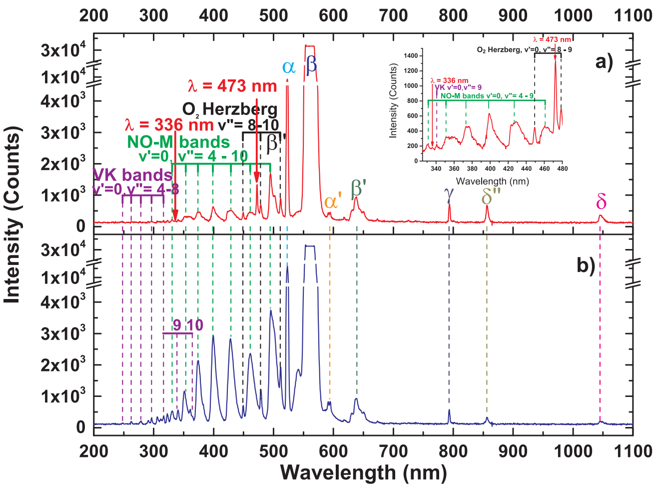

Fig. 2. Spectra taken by the Ocean Optics Spectrometer during the most intense flashes during the destruction of the samples prepared from different gas mixtures a) $[D_2]$:$[N_2]$:[He] 1:2000:100,000 (red), b) N_2:He 1:100 (blue). Each spectrum was accumulated during a period of 0.5 s. Features at $\lambda = 336$ and 473 nm are shown in the inset.

integrated spectra of the deuterium-nitrogen-helium sample.

However, the spectra of the largest flashes obtained during the destructions of both of these samples have more common features (see Fig. 2). All of the previously mentioned bands observed in the integrated spectra of the nitrogen-helium sample are present in the spectra of the largest flashes of both samples. Also the δ, δ''-groups of N atoms appeared in the spectra of both samples. Additional bands at $\lambda = 336$ and $\lambda = 473$ have appeared in the spectra of deuterium-nitrogen–helium sample (see Fig. 2a). These bands preliminarily were assigned to the emission of ND radicals, corresponding to transitions $A^3\Pi_i \rightarrow X^3\Sigma^-$ and $b^1\Sigma^+ \rightarrow X^3\Sigma^-$, respectively [23].

To understand when the emission of ND radicals appears during the destruction of the samples, we studied the dynamics of the spectra. Experiments were performed with simultaneous registration of the spectra in the range 200–1100 nm with the Ocean Optics spectrometer and in the range 900–1650 nm by the Avantes spectrometer. In Fig. 3 we combined the spectra obtained by both spectrometers. Fig. 3a shows the dynamics of the luminescence spectra in the range 200–1650 nm during the destruction of the deuterium-nitrogen-helium sample. From this figure we can conclude that in the spectra of most of the flashes, only α and δ-groups of N atoms and β-group of O atoms and the bands at $\lambda \sim 1170$ nm are present. However during the largest flashes the maximum number of molecular and atomic bands as well as the bands of the ND radical at $\lambda \sim 336$, 473, and 1170 nm are present, as can be seen in Fig. 3b. This was the first time in which the emission of the band at $\lambda = 1170$ nm was observed (shown in Fig. 3a and 3b) during the destruction of molecular nitrogen nanoclusters containing stabilized nitrogen and deuterium atoms.

To obtain further support that the emissions of the bands at $\lambda = 336$, 473, and 1170 belong to the ND radical, we performed similar studies of a nitrogen-helium sample prepared from gas mixture $[N_2]$:[He] = 1:100. Fig. 4a shows the dynamics of the luminescence spectra during the destruction of the nitrogen-helium sample in the spectral range 200–1100 nm. From this figure we can see that intensity of the α-group of N atoms increased with time and at the end of the destruction in the large flashes, many atomic and molecular bands are present. In bright flashes the δ-group of N atoms, β-group of O atoms, and NO bands are present. The spectra of the largest flash from the nitrogen-helium sample is shown in Fig. 4b. There are *no* bands from the ND radical in this spectrum. As can be seen from the spectra of the largest flash shown in Fig. 4a, there are only bands, corresponding to δ, δ', and δ'''-groups of N atoms, present in the NIR region of this spectrum. Therefore we can conclude that in the absence of deuterium in the gas mixture used for sample preparation the bands at $\lambda = 336$, 473, and 1170 are also absent. This provides additional evidence for the assignment of these bands to emission of the ND radical. Fig. 5 shows the comparison of the spectra in the region 950–1650 nm for deuterium-nitrogen-helium and nitrogen-helium samples obtained during their destruction. There is no emission near $\lambda = 1170$ nm in the spectrum of the nitrogen-helium sample. At the same time the emission of N^- anion at $\lambda = 793$ (γ-line) is present in the spectrum of the nitrogen-helium sample (Fig. 4b). This allows us to conclude that the band at $\lambda = 1170$ nm belongs to the ND radical. In the Figs. 6a and 6b the time dependences of the emission intensities of the α and δ-groups of N atoms and the bands at $\lambda = 336$ nm, 473 nm and 1170 nm are shown.

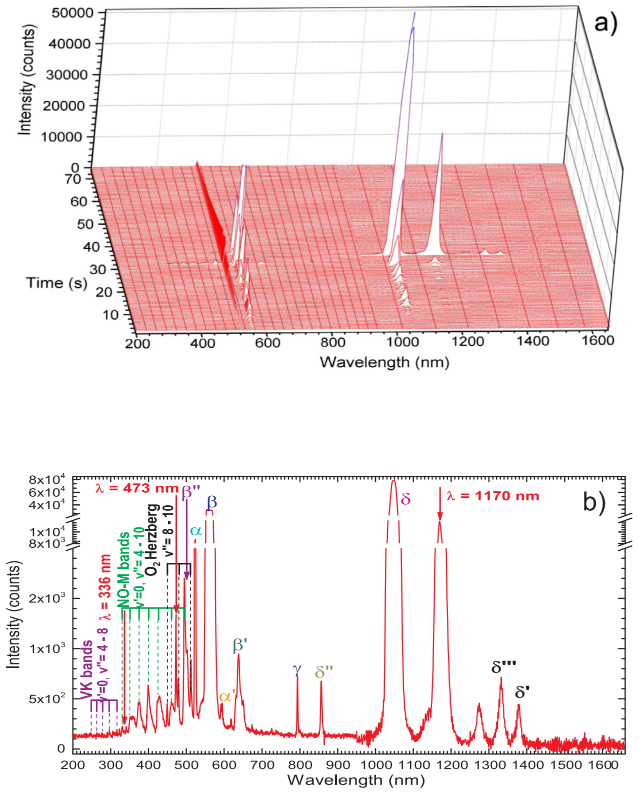

Fig. 3. Spectra obtained during the destruction of the sample prepared from the gas mixture $[D_2]$:$[N_2]$:[He] = 1:2000:100,000. a) Dynamics of the luminescence spectra. b) Spectrum of the brightest flash with identifications. Spectra were obtained by the Ocean Optics spectrometer in the range 200–1100 nm, and by Avantes spectrometer in the range 900–1650 nm. Each spectrum was accumulated during period of 500 ms.

4. Discussion

The ensembles of molecular nitrogen nanoclusters with stabilized nitrogen atoms contain high densities of stored chemical energy [10–12]. The nanoclusters form a porous structure inside superfluid helium. The pores just after preparation of the samples are filled with liquid helium [7–9]. The stabilized atoms reside mostly on the surfaces of nanoclusters [10,11]. The average concentration of nitrogen atoms in the sample was of order 10^{19} cm^{-3} [10,14], and the local concentrations of nitrogen atoms in the nanoclusters were of order 10^{21} cm^{-3} as determined from the width of electron spin resonance spectra [10]. The samples also contained three or four orders of magnitude smaller concentrations of oxygen and deuterium atoms. These porous structures were stable while immersed in bulk superfluid helium. Removing liquid helium from the porous structures led to a collapsing of the pores,

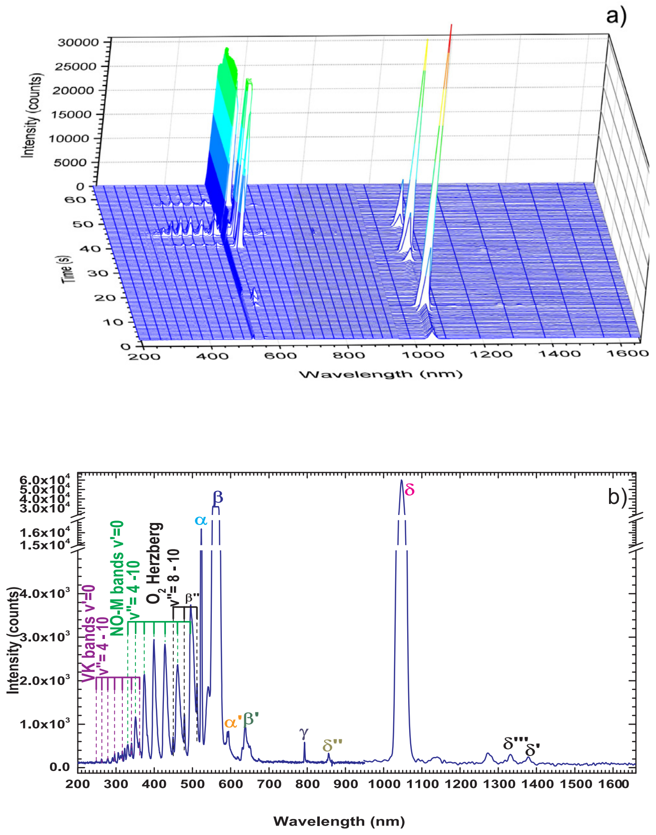

Fig. 4. Spectra obtained during the destruction of the sample prepared from the gas mixture N_2:He = 1:100. a) Dynamics of the luminescence spectra. b) Spectrum of the brightest flash. Spectra were obtained by the Ocean Optics spectrometer in the range 200–1100 nm, and by Avantes spectrometer in the range 900–1650 nm. Each spectrum was accumulated during period 500 ms.

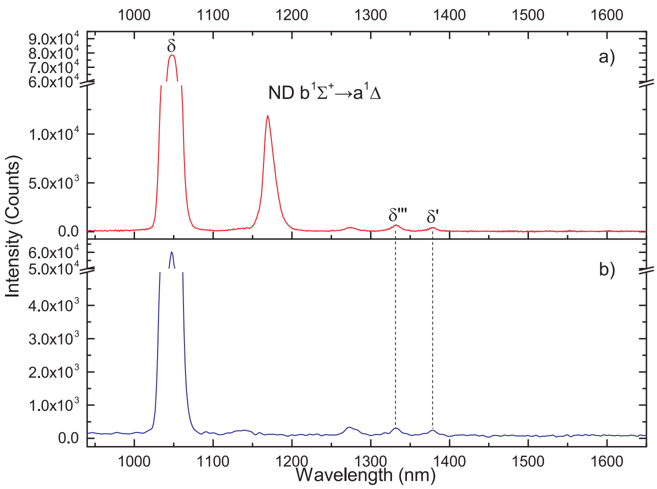

Fig. 5. Comparison of the spectra taken in the range 940–1650 nm by the Avantes Avaspec Spectrometer during destruction of the samples prepared from different gas mixtures a) $[D_2]$:$[N_2]$:[He] 1:2000:100,000 (red), b) N_2:He 1:100 (blue). Each spectrum was accumulated during a period of 0.5 s.

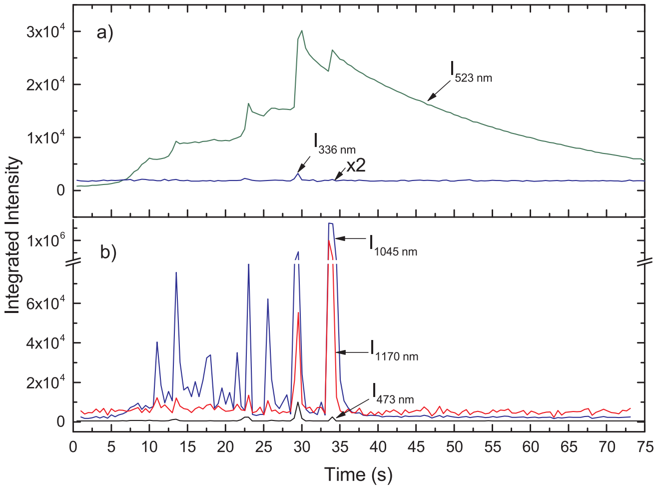

Fig. 6. a) Comparison of integrated intensities behavior in time of the α-group emission of N atoms (green line) and the emission of ND radicals at λ = 336 (blue line). b) Comparison of integrated intensities behavior in time of the δ-group emission of N atoms (blue line) and the emission at λ = 473 (black line) and 1170 nm (red line) of ND radicals.

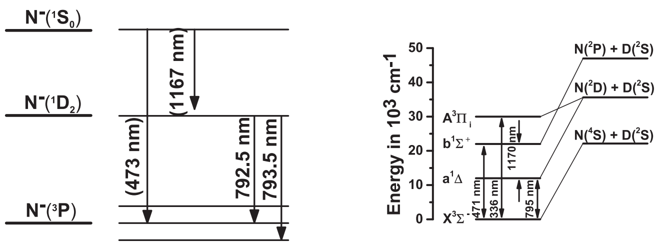

Fig. 7. a) Energy diagram for N^- anions. [28,29] b) Energy diagram for NH (ND) radicals [30].

resulting in approaching and, collisions of nanoclusters which initiated chemical reactions of atoms residing on the surfaces of the nanoclusters. Previously, the thermoluminescence during the destruction of molecular nitrogen nanoclusters containing nitrogen and oxygen atoms were studied. The well-known α, α'-groups of N atoms, the β, β', β''-groups of O atoms, the VK-bands of N_2 molecules, the M- and β-bands of NO molecules and the second Herzberg bands of O_2 molecules were observed in these studies [17–20]. The addition of hydrogen or deuterium in nitrogen-helium gas mixtures used for sample preparation led to the appearance of the band at λ = 360 nm of polymeric nitrogen N_4 and two bands at $\lambda \sim$ 336 and 473 nm, which were preliminarily assigned to the emission of the ND radical in the thermoluminescence spectra during sample destruction [23]. The only remaining problem was with the accuracy of the assignment of the band at λ = 473 nm to the $b^1\Sigma^+ \to X^3\Sigma_-$ transition of ND radical, because the alternative interpretation for this band could perhaps be the $^1S_0$-$^3P_{1,2}$ transition of N^- anion (see Fig. 7a).

Fig. 7a shows the energy diagram for the three lowest energy terms of the N^- anion. The transition ($^1S_0 \to {}^3P_1$) had never been observed previously for the N^- anion. If this assignment would be correct, we can find the position of the third transition, $^1S_0$–$^1D_2$, of the N^- anion shown in Fig. 7a because the characteristics of the γ-line (at λ = 793 nm) corresponding to the $^1D_2$–$^3P_{1,2}$ transition of N^- anion is well known [22]. The result of the calculation for the $^1S_0$–$^1D_2$ transition of N^- anion provides a value λ = 1167 nm. This simple analysis motivates us to perform experiments with simultaneous registration of thermoluminescence spectra during the destructions of deuterium-nitrogen-helium samples in the UV, visual and near infrared, and compare these spectra with that obtained for "pure" nitrogen-helium samples for which the observation of the N^- anion bands might be expected.

Figs. 1a and b shows that there are *no* bands at λ = 336 and 473 nm in the integrated spectra of thermoluminescence of the two samples studied. Both these bands together with the band at λ = 1170 nm are present in the spectra of the most intense flashes during the destruction of deuterium-nitrogen-helium samples (see Fig. 3b). Therefore we assigned these three bands to the emission of the ND radical. Fig. 7b shows the energy diagram for the NH(ND) radicals. Bands at λ = 336, 471 and 1170 nm were assigned to the transitions $A^3\Pi_i \rightarrow X^3\Sigma^-$, $b^1\Sigma^+ \rightarrow X^3\Sigma^-$ and $b^1\Sigma^+ \rightarrow a^1\Delta$, respectively, of ND radicals. The formation mechanism for excited states of ND radicals could be a result of recombination reactions of nitrogen atoms in excited states $N(^2D)$ and $N(^2P)$ with deuterium atoms in the ground state.

$$N(^2D) + D(^2S) \rightarrow ND(A^3\Pi_i) \tag{1}$$

$$N(^2P) + D(^2S) \rightarrow ND(b^1\Sigma^+) \tag{2}$$

The presence of substantial quantities of $N(^2D)$ and $N(^2P)$ atoms in nanoclusters is supported by the observation of intense emission of α- and δ-groups where the lifetime of α-group is ~ 30 s and the lifetime of δ-group is ~1 ms in solid molecular nitrogen [31].

As a consequence of these reactions the emissions from $A^3\Pi_i$ state which corresponds to the bands at $\lambda = 336$ nm should correlate with the emission of α-group of nitrogen atoms, transition ^2D–^4S. The emission from $b^1\Sigma^+$ state of ND radical which corresponds to the bands at $\lambda = 473$ nm and 1170 nm should correlate with the emission of the δ-group of nitrogen atoms, transition ^2P–^2D. Figs. 6a and b show that intensities of ND radical bands do correlate with the intensities of the corresponding α and δ-group emissions of nitrogen atoms only in the largest flashes.

5. Conclusions

1. Comparison of the spectra obtained during the destruction of nitrogen-helium and deuterium-nitrogen-helium samples provided evidence for assignment of the bands observed in the luminescence of deuterium-nitrogen-helium samples at $\lambda = 336$, 471, and 1170 to the emission of ND radicals with corresponding transitions $A^3\Pi_i^+ \rightarrow X^3\Sigma^-$, $b^1\Sigma^+ \rightarrow X^3\Sigma^-$, $b^1\Sigma^+ \rightarrow a^1\Delta$.
2. These ND bands were observed during the final stages of destruction of the samples. The processes of recombination of excited metastable nitrogen atoms with deuterium atoms in ground state are responsible for the appearance of excited states of the ND radical.

Acknowledgments

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