Quantum Electronics **46** (8) 713–718 (2016)

INTERACTION OF LASER RADIATION WITH MATTER DOI: 10.1070/QEL16094

Influence of laser-induced breakdown on the fragmentation of gold nanoparticles in water

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Abstract. **The process of fragmentation of gold nanoparticles in water exposed to nanosecond IR laser radiation (1060–1070 nm) is studied. The behaviour of the size distribution and extinction spectrum of nanoparticles is investigated at different experimental parameters. The dependences of the size distribution maximum position and distribution width on the laser pulse duration and the corresponding characteristics of the laser plasma, arising on the nanoparticles, are analysed. The results are explained by the plasma influence on the laser radiation absorption by the colloidal solution.**

Keywords: *laser-induced breakdown, fragmentation of nanoparticles in liquid.*

1. Introduction

The interaction of pulsed laser radiation with nanoparticle colloidal solutions has been a subject of both theoretical and experimental studies for the last two decades. As a rule, this interaction leads to the reduction of the nanoparticle size, also referred to as fragmentation [1–4]. The size of nanoparticles decreases with increasing number of laser pulses absorbed by the nanoparticle. The maximum of the nanoparticle size distribution in this case is shifted towards smaller sizes.

To date, there is no clear description of the process of the laser-induced nanoparticle fragmentation in liquids. However, there are multiple works devoted to the study of possible mechanisms, whose combination determines the interaction of laser radiation with colloidal solutions of nanoparticles. All these mechanisms imply the heating of the nanoparticle up to the melting temperature. In this case, the fragmentation can occur due to hydrodynamic instabilities arising at the melted metal–surrounding liquid interface [5, 6], photothermal processes [7], or Coulomb explosion. In the last-named case, a large number of electrons are emitted from the nanoparticles, which leads to their ionisation followed by the spontaneous fragmentation due to Coulomb repulsion. Kamat et al. [8] used this model to interpret the fragmentation of silver nanoparticles under the impact of picosecond laser radiation with a wavelength of 355 nm, since the solvated electrons were observed in the working liquid during irradiation. The authors of Refs [9, 10] also observed solvated electrons under exposure of gold nanoparticles to nanosecond laser radiation in an aqueous solution of sodium lauril sulphate. According to the results of these papers, under the action of radiation with a wavelength of 335 nm the efficiency of fragmentation is higher than under the action accompanied by the excitation of a plasmon resonance in gold nanoparticles. The observed effect is explained by the fact that during the intraband excitation, the 'bleaching' of the plasmon resonance occurs, which leads to the reduction of the effective absorption coefficient. In the case of an interband transition excitation, the relaxation time of electrons is smaller, and practically no reduction of absorption occurs. The authors of papers [11, 12] detected positive ions of gold using the mass-spectrometry methods. As a mechanism of their production, the thermionic emission due to the excitation of the interband transition was proposed.

It is worth noting that in all mentioned papers the key role is played by the emission of electrons from the nanoparticles (independent of its particular mechanisms). It is known that under certain experimental parameters, the formation of plasma with a critical concentration of free electrons is possible, and this plasma can play a significant role in the fragmentation process. Note that the absorption maximum of the majority of nanoparticles is located in the visible and UV ranges of the spectrum [13]. Hence, in the case of IR laser sources the radiation absorption is mainly due to the absorption of the plasma that appears around the nanoparticles. We should also emphasise that the plasma plume formation requires from tens to hundreds of picoseconds [14, 15]. Therefore, in using laser sources with a pulse duration of tens or hundreds of nanoseconds, a certain part of the laser pulse is absorbed directly by the plasma plume [16].

In spite of the presented facts, the role of laser plasma in the nanoparticle fragmentation process in liquid is scarcely explored. It is implied that the plasma plume can be a source of embryonal clusters from which the nanoparticles can be formed by aggregation at the plume–liquid interface [17].

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Received 31 March 2016; revision received 6 June 2016
Kvantovaya Elektronika **46** (8) 713–718 (2016)
Translated by V.L. Derbov

However, the influence of plasma on nanoparticles already existing in the colloidal solution remains unexplored. As mentioned above, the plasma formation can essentially affect the absorption of laser radiation. The subsequent transfer of the plasma energy to the nanoparticles can occur via several mechanisms, such as the direct absorption of the plasma radiation, heat exchange, etc. The fragmentation of nanoparticles in this case is accelerated due to the more efficient transfer of the laser radiation energy to the nanoparticles.

The aim of the present paper is to study experimentally the influence of laser-induced breakdown on the process of fragmentation of gold nanoparticles in water. We analyse the dependences of the size distribution function of nanoparticles on different experimental parameters, as well as the properties of the plasma plume.

2. Experimental setup

The initial colloidal solution of gold nanoparticles was obtained using the method of laser ablation in liquid [18]. As a source of radiation at the wavelength 1060–1070 nm we used an ytterbium fibre laser with a pulse duration of 70 ns, a pulse repetition rate of 20 kHz and a pulse energy of 1 mJ. The laser radiation was focused onto the surface of a gold target by means of a F-Theta objective (with a focal length of 207 mm). As a working liquid, we used deionised water. The laser fluence at the target surface amounted to nearly 13 J cm^{-2} (estimated by the size of a heat-affected zone on the target). The laser beam was scanned by means of a galvo-optical system. The concentration of gold nanoparticles in the resulting colloid amounted to $\sim 10^{13}$ cm^{-3}. This quantity is estimated based on the size distribution function of the particles and on the target mass defect [5]. The typical rate of nanoparticles generation (0.5 mg min^{-1}) decreased with time due to increasing absorption and scattering of radiation by the colloid particles. To stabilise the particles after their production, a certain amount of polyvinyl pyrrolidone (PVP) (0.5 mg mL^{-1}) was added.

The colloidal solution of gold nanoparticles in the absence of the target was subsequently irradiated by an ytterbium fibre laser (the wavelengths 1060–1070 nm, the mean power 20 W) with a variable pulse duration (4–200 ns, the pulse repetition rate 500–20 kHz, the pulse energy 0.04–1 mJ). To study the temporal plasma characteristics, we used Thorlabs DET10A photodiodes (the rise time 1 ns), the signal from which was fed to a GW Instek GDS 72204E oscilloscope (the transmission bandwidth 200 MHz). The schematic of the experimental setup for the nanoparticle fragmentation is presented in Fig. 1. The scattered laser light was cut off from the plasma radiation using a cutoff optical filter installed in collimator (*6*).

The extinction spectra of the colloidal solutions of nanoparticles before and after the fragmentation were measured using an Ocean Optics fibre spectrometer. The behaviour of the size distribution function of the nanoparticles was studied using a CPS Instruments 24000 measuring disc centrifuge.

3. Experimental results

It is known that the focusing of sufficiently intense laser radiation into a liquid leads to its breakdown, i.e., the formation of plasma surrounded by a gas-vapour bubble. The

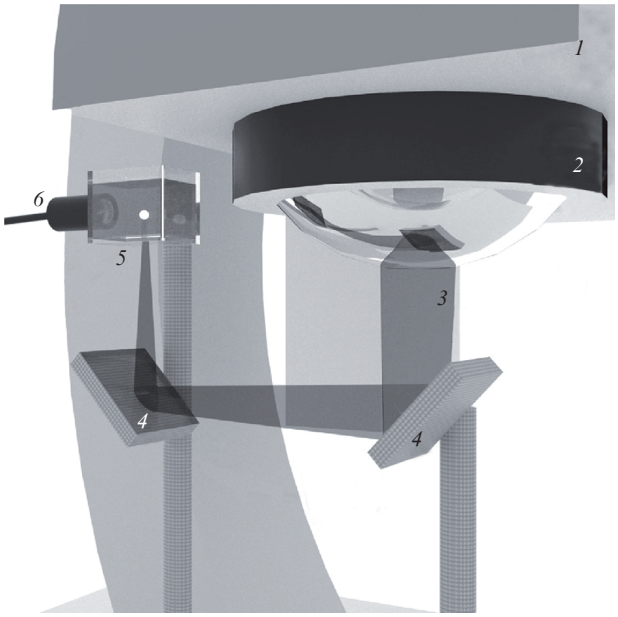

Figure 1. Schematic of the experimental setup for fragmentation of gold nanoparticles in water:
(*1*) scanning head; (*2*) focusing objective; (*3*) laser beam; (*4*) dielectric mirrors; (*5*) silica cell with the colloidal solution; (*6*) optical fibre with collimator.

breakdown threshold intensity is determined by such laser radiation parameters as the wavelength, pulse energy and pulse duration [14]. Besides the mentioned properties of the radiation source, the occurrence of the breakdown is also determined by the properties of the liquid itself. In particular, the presence of impurities can essentially reduce the breakdown threshold intensity. It was shown that if the role of impurities is played by nanoparticles, then the plasma properties depend on both their size and material [19, 20].

In all the experiments of the present paper, we used gold nanoparticles with an average transverse size of about 20 nm. The longitudinal size varied from 20 to 100 nm in analogy with the nanoparticles used in Ref. [21]. It is worth noting that under our experimental conditions the breakdown was not observed in the absence of nanoparticles. The typical oscillograms of the laser pulse and the plasma emission are presented in Fig. 2.

According to Fig. 2, in both cases the time profile of the plasma virtually corresponds to the profile of the pulse. The delay between the beginning of the laser pulse and the appearance of the plasma signal is such that the plasma plume appears in the maximum of the laser pulse and decays in a few nanoseconds after the end of the pulse. This fact means that plasma plume is supported by the laser pulse via the radiation absorption. The plasma lifetime in all cases is nearly equal to the laser pulse duration. Note, that the duration in all cases is determined as the signal width at a half-maximum level.

It should be emphasised that for the pulse durations of 14–100 ns, the initiation of the breakdown is sporadic. To compare these regimes with the effect of the 200 ns pulsed radiation, when the stable emission from the plasma is observed, we measured the amplitude distributions of the glow signal from a photodiode (Fig. 3). For brevity, only the data for 100 and 200 ns are presented.

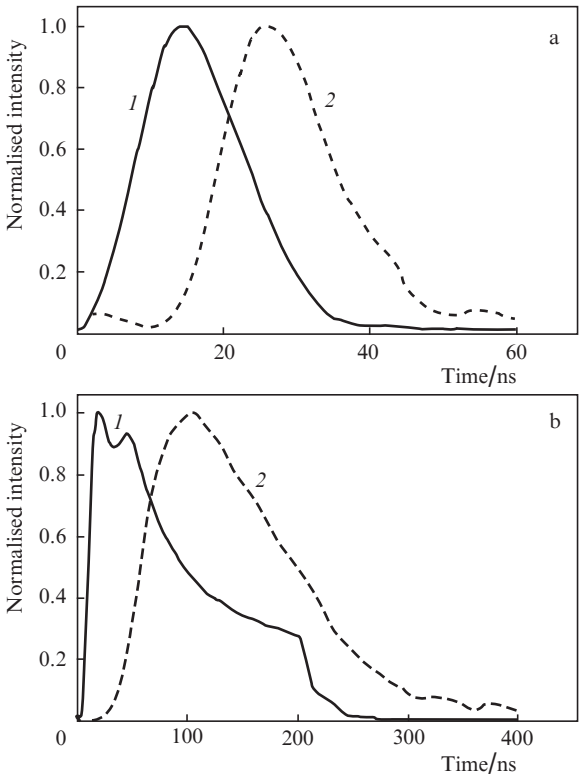

Figure 2. Typical oscillograms of (*1*) the laser pulse and (2) plasma glow. The pulse duration is (a) 14 and (b) 200 ns, and the pulse energy is 0.16 and 1 mJ, respectively.

For the pulse duration of 100 ns only low-amplitude breakdown events take place. The distribution of the plasma glow signal for the 200 ns regime possesses a well-defined maximum, corresponding to brighter breakdowns. At the same time, for 100 ns there is no such maximum and the number of counts monotonically decreases with increasing amplitude.

To study the effect of the laser-induced breakdown on the process of nanoparticle fragmentation, in each of the described regimes (14–200 ns) a series of exposures was performed, in which the exposure time varied from 0.5 to 4 min. For each exposure, a new portion (2 mL) of the initial colloidal solution was used.

The typical evolution of the size distribution function of the nanoparticles is presented in Fig. 4a and the corresponding evolution of the colloid extinction spectrum is shown in Fig. 4b.

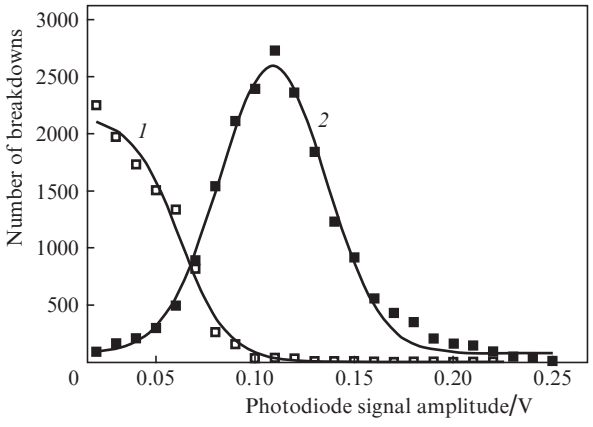

Figure 3. Amplitude distributions of the photodiode signal for the pulse durations (*1*) 100 and (*2*) 200 ns. The acquisition time is 20 s, the pulse energy is 0.5 and 1 mJ, and the repetition rate is 40 and 20 kHz, respectively.

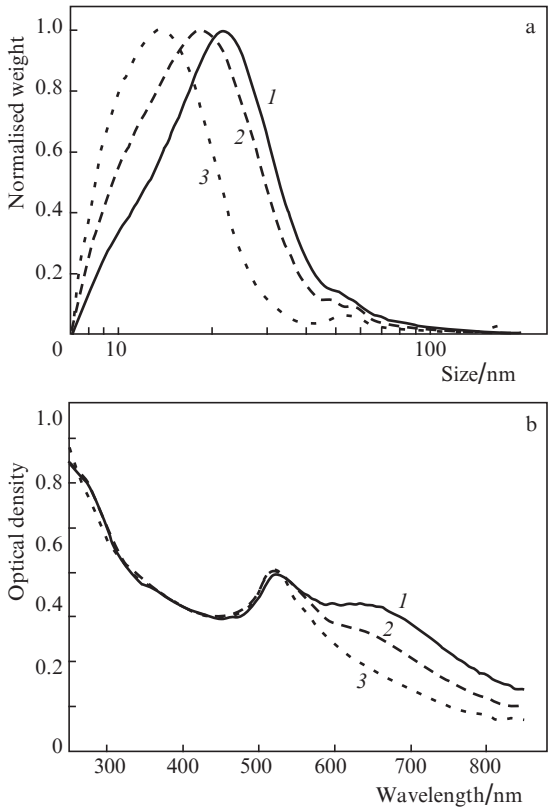

Figure 4. (a) Evolution of the size distribution function of gold nanoparticles and (b) extinction spectrum: initial colloid (*1*) after irradiation during 0.5 (*2*) and 4 min (*3*). The laser radiation wavelength is 1060–1070 nm, the pulse duration is 200 ns, the pulse energy is 1 mJ, and the pulse repetition rate is 20 kHz.

According to Fig. 4, under exposure to 200 ns radiation the initial maximum is shifted from 21.7 to 13.5 nm with a simultaneous reduction of its width. The decrease in the colloidal solution extinction in the red part of the spectrum (about 650 nm) corresponds to the fragmentation of elongated particles with the aspect ratio 1:2 and more [22]. The observed change in the size distribution function of nanoparticles under exposure to laser radiation is typical for such experimental conditions and was repeatedly described in previous papers [23, 24]. In this case, the subject of major interest is to compare the evolution for various pulse durations of laser radiation (Fig. 5).

According to the presented data, for a pulse duration of 200 ns the fragmentation is significantly more efficient, so that the mean size of the nanoparticles amounts to 13.5 nm, the width of the maximum being 13 nm. This can be due to the greater probability of the plasma formation for laser pulses having a greater duration, observed experimentally (see Fig. 3).

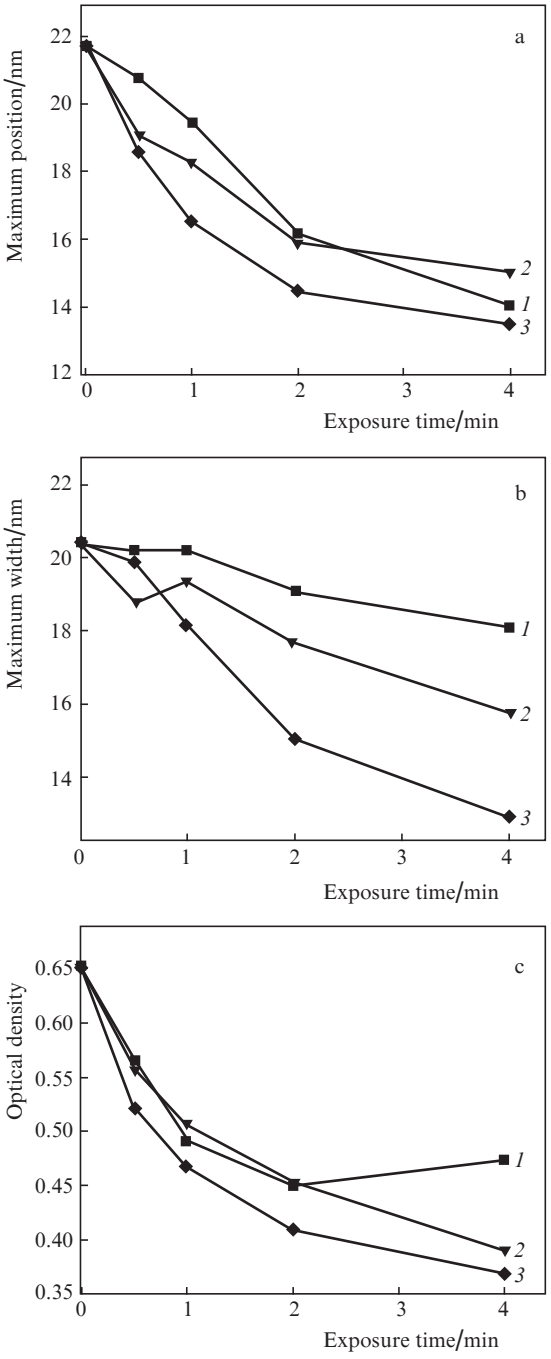

Figure 5. Evolution of (a) the maximum position of the size distribution function, (b) the maximum width and (c) the extinction at the wavelength 650 nm for the laser pulse duration (*1*) 14, (*2*) 100, and (*3*) 200 ns at pulse energies of 0.16, 0.5 and 1 mJ, respectively.

4. Discussion

As mentioned above, in the existing fragmentation models the influence of the laser-induced breakdown in the liquid that occurs under exposure to laser radiation of sufficiently high intensity (above 10^8 W cm^{-2}) is not taken into account. We should emphasise that under the experimental conditions of the present paper, one cannot neglect the effect of plasma on the fragmentation process. In particular, according to the estimate presented below, based on the two-temperature model [25], no significant change in the nanoparticle temperature occurs due to the direct absorption of laser radiation. The temperature of the lattice $T_{\rm lat}$ and the electrons $T_{\rm e}$ can be presented in the following form:

$$T_{\rm lat}(t) \approx T_0 + \frac{a^2 S(t)}{3\kappa}, \tag{1}$$

$$T_{\rm e}(t) \approx T_{\rm lat}(t) + \frac{S(t)}{G}, \tag{2}$$

where T_0 is the initial temperature (300 K); a is the particle radius; κ is the heat conduction coefficient of water (W m^{-1} K^{-1}); and G is the electron–phonon coupling constant (2.5×10^{16} W K m^{-3}) [26]. The absorption of the laser radiation by a particle is described by the formula

$$S(t) = \frac{\sigma_{\rm abs}}{\frac{4}{3}\pi a^3} I_0(t),$$

where $I_0(t)$ is the radiation intensity;

$$\sigma_{\rm abs} = 12\pi a^3 \frac{\omega}{c} \frac{n_0^3 \varepsilon''}{(\varepsilon' + 2n_0^2)^2 + \varepsilon''^2}$$

is the nanoparticle absorption cross section [27]; ω is the angular frequency of the radiation; c is the speed of light; n_0 is the refractive index of the medium (equal to 1.33 for water); and $\varepsilon = \varepsilon' + i\varepsilon''$ is the permittivity of gold (equal to $-43.926 + 4.185$i for the wavelength 1065 nm). Using expressions (1) and (2) one can show that for the radiation intensity of 1.6×10^{11} W m^{-2} the change in the temperature of a gold nanoparticle having a diameter 20 nm amounts to less than 5 K. Thus, one can see that the description of the process based on the two-temperature model does not provide appropriate correspondence to the experimental data, namely, the observed fragmentation of nanoparticles.

In the above temperature estimate, the so-called field enhancement on the nanoparticles is taken into account. This effect arises due to the interaction between the photons of the incident radiation and the collective oscillations of electrons at the surface of the particle. The expression for the enhancement coefficient looks as [28]

$$\eta \equiv \frac{|E_2|^2}{|E_0|^2} \approx \left|1 + 2\frac{\varepsilon - n_0^2}{\varepsilon + 2n_0^2}\right|,$$

where $|E_2|^2$ and $|E_0|^2$ are squared absolute values of the field strength inside and outside the particle, respectively. For a gold nanoparticle in water, the coefficient of field enhancement approaches 12 at the wavelength 1065 nm. However, as follows from the above estimates, at the incident radiation intensity 1.6×10^{11} W m^{-2} this is insufficient to heat the nanoparticle.

Is should be noted that all the above estimates are valid for an individual gold nanoparticle in water. In the experiment, we used a colloidal solution with a concentration 10^{13} cm^{-3}, which leads to the necessity to take the possible interaction between the particles into account. The rate of their collision as Brownian particles [23] is

$$\vartheta \approx 6D\sigma^2 n^3,$$

where $D \approx 10^{-7}$ cm^{2}s^{-1} is the diffusion coefficient; $\sigma = 4\pi R^2$ is the geometric cross section of the particles having a radius R; and n is their concentration. Thus, the collision frequency amounts to nearly 1.2×10^{21} m^{-3} s^{-1}. If we present the waist region of the laser beam as a cylinder with the height h = 100 μm and the diameter d = 100 μm, then the number N of the collisions of the particles that occur in the cylinder during a laser pulse can be estimated as:

$$N = \vartheta V\tau = \frac{\pi}{4}\vartheta h d^2\tau,$$

where $V = \pi h d^2/4$ is the volume of the waist region; and τ is the laser pulse duration. From these data we obtain $N \approx 2 \times 10^5$ for τ = 200 ns.

According to the presented estimate, the collisions of nanoparticles do occur in the beam waist region during the laser pulse. During a certain time a pair of colliding particle or particles separated by a small distance can be considered as a dimer in the field of external laser radiation. It should be noted that the interaction of electrons of nanoparticles with the external field occurs at the time scale of a few femtoseconds or tens of femtoseconds [29], and the particles move in the colloid with the thermal velocities. Therefore, for the processes related to the absorption of the radiation by electrons, one can neglect the motion of nanoparticles in the colloid. The dimers similar to those appearing for a short time during the collision of nanoparticles, in recent years have become a subject of numerous studies due to the field enhancement that arises in them, exceeding by many times the field enhancement on individual particles [30]. According to different data, the field enhancement coefficient for dimers can vary from a few tens to a few thousands [29, 31–33]. Such properties of the dimers allow using them for detecting the signal from individual molecules in the surface-enhanced Raman spectroscopy [34, 35].

In the present paper, the formation of gold nanoparticle dimers is expected to provide a laser radiation field enhancement, sufficient for the emission of electrons from the particle surface. Note that the particular mechanism of the emission is still unknown. If the particles are heated to a few thousand kelvins, then the key mechanism is the thermionic emission. [25]. Otherwise, the field emission [36] or the multiphoton ionisation of gold atoms [25] occurs. The emitted electrons absorb the laser radiation due to the inverse bremsstrahlung absorption [37]. Upon reaching the critical energy of the electrons $E_c \approx 1.5\Delta$, where Δ is the ionisation potential of water molecules (6.5 eV [38]), the electron avalanche is initiated due to impact ionisation [39, 40]. Thus, in the colloidal solution the plasma appears at the gold nanoparticles under the radiation intensities significantly lower than the threshold intensity of the laser-induced breakdown [14]. Such plasma is referred to as nanoplasma because of its localisation around the nanoparticles [15]. It is frequently used in medicine for point impact of laser radiation on tissues [41].

After the appearance of plasma, the liquid around the nanoparticle is heated which, in turn, leads to the formation of a vapour-gas bubble. Its maximal radius R_m and lifetime t_r can be estimated using the model presented in Ref. [42]:

$$R_m \approx r_0 (P_{g0}/P_\infty)^{1/3\gamma}, \quad (3)$$

$$t_r \approx 1.83 R_m (\rho_\infty/P_\infty)^{1/2}, \quad (4)$$

where r_0 is the initial radius (in the present case, the radius of the region where the plasma is localised); P_∞ is the atmospheric pressure; γ is the adiabatic index (equal to 5/3 in this case [43]); ρ_∞ is the water density under the normal conditions;

$$P_{g0} = P_\infty + C_\infty \left(\frac{\rho_c E_b}{8\pi r_0^3}\right)^{1/2}$$

is the initial pressure of gas in the bubble; C_∞ is the velocity of sound in water under the normal conditions; ρ_c is the critical density of water (322 kg m^{-3}); and E_b is the bubble energy (nearly 3% of the plasma energy [42]). Assuming the initial size of the bubble to be comparable with the size of the nanoparticle (20 nm), we obtain $R_m \approx$ 500 nm, $t_r \approx$ 100 ns. Under irradiation of the colloidal solution, an increase in the pulse duration leads to an increase in the number of nanoparticle collisions in the waist region, to the formation of nanoscale plasma on them, and to subsequent formation of vapour-gas bubbles. The increasing number of bubbles due to the growth of the number of collisions leads to the increased probability of their fusion. One can show that for $N = 2 \times 10^5$ the total volume of the bubbles having the radius $R_m \approx$ 500 nm constitute a considerable part of the waist region volume (the cylinder with the diameter 100 μm and the height 100 μm).

Thus, an increase in the pulse duration leads to an increase in the probability of fusing the bubbles that contain nanoplasma and subsequent initiation of the laser breakdown. In this case, the absorption of radiation by the colloidal solution increases up to 90% [44]. As a result, part of energy is transferred to the plasma radiation, which is known to be approximated by the continuum in the visible spectral range [14]. Therefore, the effective coefficient of absorption of such radiation by the nanoparticles is much greater than that at the wavelength of the laser radiation. From this fact, we can draw a conclusion that one of the ways of transferring the plasma energy to nanoparticles is the plasma radiation. On the other hand, since the plasma initially appears around the nanoparticles, the heat transfer can also be the mechanism. For accurate description of the process of energy transfer from plasma to nanoparticles further experimental and theoretical studies are needed.

5. Conclusions

We studied the process of fragmentation of gold nanoparticles in water exposed to nanosecond IR laser radiation. It was shown that for a sufficiently low (1.6×10^{11} W m^{-2}) intensity of laser radiation, the maximum of the size distribution function is shifted towards smaller sizes. This effect is not explained by the conventional two-temperature model, which yields the temperature of nanoparticles insufficient for their melting. Besides that, we studied the dependence of the distribution function behaviour on the laser pulse duration. It was shown that an increase in the pulse duration leads to the growth of the fragmentation rate. The results were explained based on the assumption that during the collision of the particles the laser radiation field enhancement occurs at them that gives rise to the plasma formation at the essentially lower intensity than the breakdown threshold in the pure liquid. For the pulse duration of 200 ns the number of collisions in the beam waist region achieves the critical value, sufficient to fuse the plasma, arising at the nanoparticles, into a plasma plume,

corresponding to the liquid breakdown. The rate of nanoparticle fragmentation in this case increases as a result of more efficient absorption of the laser radiation by the colloidal solution.

Acknowledgements. The work was partially supported by the President of the Russian Federation (SP-753.205.2 and Grant No. MK-1494.2015.2) and the Russian Foundation for Basic Research (Grant Nos 15-02-04510, 15-32-20926, 16-02-01054).

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