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Alkali-metals flash duration during multibubble sonoluminescence from aqueous solutions of their salts determined using time-correlated single photon counting

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The time-correlated single photon counting method (TCSPC) was used for determining the flash duration of alkali-metal atomic lines of Na (589 nm), Li (671 nm), K (767 nm), Rb (780 nm) during multibubble sonoluminescence from alkali-metal chloride salts aqueous solutions. Measurements showed the values of 5 to 40 ns for different metals and different experimental conditions. Both the increase in the atomic mass by 12 times in a series Li, Na, K, Rb and the decrease in the excitation energy of the metal by a third in the series Na, Li, K, Rb resulted in the flash duration increasing approximately twofold. For all metals the flash duration was approximately twice as long in the narrow range of $\Delta\lambda \sim 5$ nm, which included the spectral line, as compared with the wide range of $\Delta\lambda \sim 200$ nm around the line. The increase of concentration of Na, K, Rb salt solutions from 1 to 3 M resulted in a decrease in flash duration by about a quarter. The addition of a surfactant (sodium dodecyl sulfate) to metals salt low concentrated solutions (2 mM) led to increase of flash duration approximately twice in comparable to high concentration solutions for all metals. Possible explanations of the results are proposed.

Keywords: sonoluminescence, metal flash duration, correlation method.

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Introduction

The appearance of atomic lines of metals in the sonoluminescence (SL) spectra of solutions of their non-volatile compounds is still an open issue, as, indeed, are most aspects of this multifactorial phenomenon. Interest in aqueous solutions of alkali metal salts is not only due to the relative simplicity of their SL spectra, but also to the wide distribution of these compounds in natural fluids, including physiological ones. Ultrasound, for example, is widely used in medicine. Its action on liquids is accompanied by the appearance of cavitation bubbles and associated cavitation effects, including weak luminescence - sonoluminescence. SL occurs in the form of very short flashes correlating with the moment of bubble collapse. Information about SL mechanisms can be provided by the duration of the emitted photon packets and their correlation with different phases of bubble dynamics, including the moment of the compression peak, when a continuum of the spectrum formed by the most energy-intensive processes is emitted. The method of time-correlated single photon counting (TCSPC), first applied to the stable SL of a single bubble, allowed determining the duration of the continuum flash 150 ps, while providing evidence for the non-thermal nature of the SL spectrum in this mode [1]. Note that we are aware of only one paper using TCSPC for multibubble system, which presents flash duration data for the integral spectrum [2]. The difficulty in obtaining such data for multibubble systems is due to the inconsistency of the flashes of individual bubbles and the small number of photons in the flash. Nevertheless, the software-based temporal correlation method we have developed has been successful in extracting flash data from the „of an average" bubble in selected spectral bands, i.e., belonging to individual emitters, and in estimating the temporal shifts between their emission [3–6]. In this paper we investigate the duration of the flashes of alkali-metal atoms emission under different experimental conditions and different recording conditions by the TCSPC method, and suggest possible explanations for the dependences obtained.

Experimental part

Figure 1 shows a schematic diagram of the experimental setup for measuring the spectra of multibubble SL (1) and photon correlation spectra (2). The multibubble SL of aqueous solutions of 1 and 3 M NaCl, KCl, LiCl, RbCl and for a range of NaCl concentrations was investigated, as well as the effect of surfactant on the flash duration of metal atoms in salt solutions of concentration 2 mM when 2 mM sodium dodecyl sulfate (SDS) was added. 0.51 solutions of salts (reagent grade) prepared in distilled water were saturated with argon in a Drexel vessel placed in a Julabo F12 thermostat. The rate of argon bubbling through the

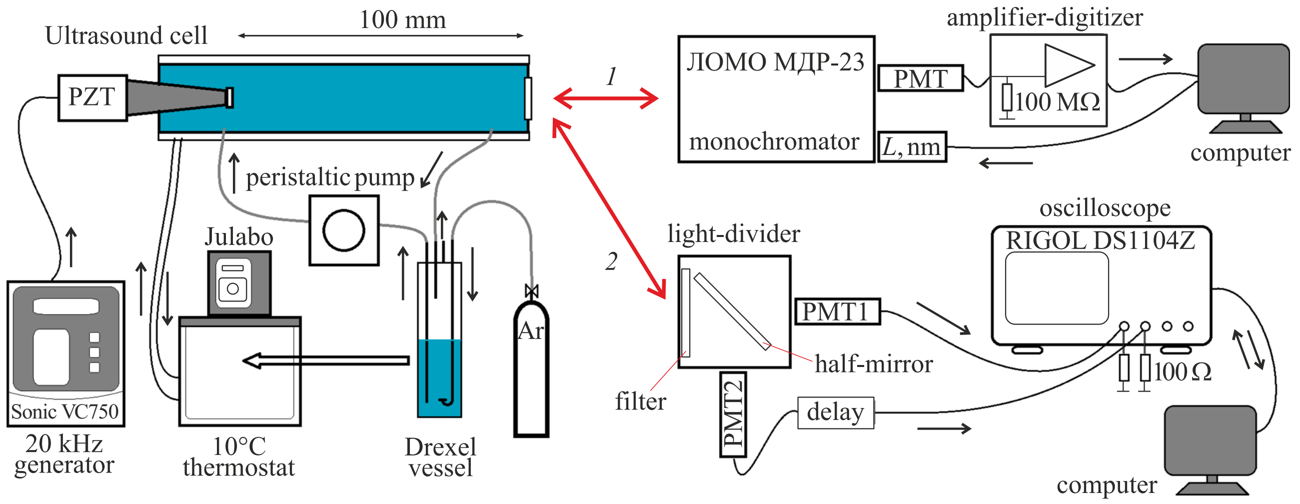

Figure 1. Diagram of the setup.

solution was 0.02 l/min. The temperature of the solutions was kept at 10 ± 1°C. The solution was continuously pumped through a thermostated stainless steel cell with a volume of 50 ml using a peristaltic pump, which was also used to create additional pressure in the cell (~ 0.2 atm), which allowed to increase the brightness of metal lines. The pumping rate of the solution was 0.12 l/min. The tip of the ultrasonic emitter was placed in one end of the cell, the tip area was 1.5 cm^2. The other end was covered with a quartz window, which was connected (1) to the input slit of a LOMO MDR-23 scanning monochromator (slit width 2.2 mm, resolution 2.9 nm) or (2) to the beam splitter of a correlation counter. The distance from the quartz window of the cell to the transmitter tip was 100 mm. Sonics Vibra Cell VC750 generator, frequency 20 kHz, power output ~ 20 W was used as the ultrasound source. The photomultiplier FEU-79 was used in the accumulation mode with a high impedance load ~ 100 MOm, the PMT sensitivity area was 300–800 nm, the maximum blaze of the diffraction grating of the monochromator was 500 nm. The SL spectra were obtained without correction for the spectral sensitivity of the system „of the PMT + grating“.

The duration of flashes was measured using a two-channel correlation counter (2) based on two FEU-79 operating in the photon-counting mode with a low-resistance load ~ 100 Om, a RIGOL DS1104Z digital oscilloscope, and a computer (detailed description is given in [3,7]). A light filter and a semi-transparent mirror were placed in the light splitter, which used three thin quartz glasses dividing the light flux into transmitted and reflected light in the ratio $\sim 70 : 30\%$. The duration of the flashes was measured in the autocorrelation mode. A 3 m long cable was used as a delay line in one of the channels. The measured flash duration value W was obtained as the full width at half maximum (FWHM) of the Gaussian curve by approximating the correlation peak. The correlation method, in our case of multi-bubble SL, gives the mean W of the bubbles [4]. „The true“ flash duration can be calculated as $W_r = ((W^2 - W_0^2)/2)^{1/2}$ taking into account a correction for the intrinsic duration W_0 introduced by the correlation counter, namely each PMT and the oscilloscope digitisation circuits [7]. We estimate the value of W_0 as ~ 1.7 ns based on the following considerations. The duration of water SL flashes under conditions similar to ours was found to be 0.25 ns [2], our measurements of water SL flashes yielded 1.8 ns [8]. Since the W of the metals measured in the present study are high (5–40 ns), the above correction is not significant for them. Flashes at different wavelengths of the spectrum were separated using broadband ($\Delta\lambda \sim 200$ nm) light filters or a monochromator ($\Delta\lambda \sim 5$ nm). A narrow-band ($\Delta\lambda \sim 11$ nm) interference light filter BP589 was also used for the Na line.

SL spectra and correlation spectra

The SL spectra in the 400–850 nm range of 3 M salt solutions are shown in Fig. 2. Significantly broadened metal lines are observed in the background of the continuum extending from IR to UV with a maximum of ~ 420 nm at a given spectral sensitivity of the „PMT+grating“ system. The doublet line structure is visible only for Rb, for the other metals the broadening of the lines does not allow us to resolve their doublet structure. The reason for the broadening is the high density of the emitting medium, which can exceed 600 amg ($1\ \text{amg} = 2.6868 \cdot 10^{19}\ \text{cm}^{-3}$) [9]. Now in the SL lines of alkali metals, in particular Na, two components are distinguished, a broad and a narrow one, when a narrow unshifted line is observed against the background of the density-broadened the line, corresponding to the line in the spectrum of the flame. The nature of the narrow component in the SL spectrum is not clear [10–12]. The short-wavelength part of the broadened component with a noticeable hump is called the blue satellite, the long-wavelength slope — the red satellite [13].

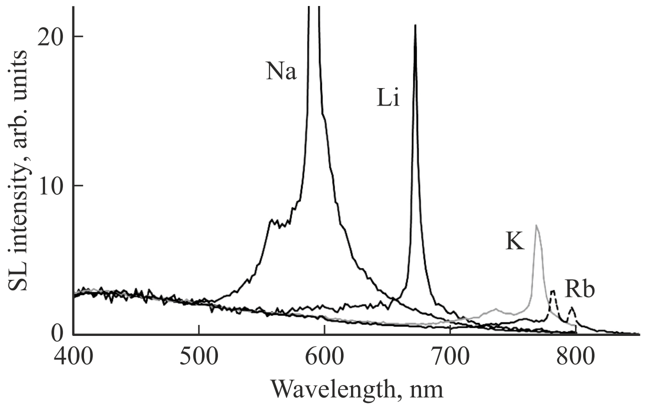

Figure 2. The SL spectra of aqueous 3 M solutions of alkali metal chlorides at Ar saturation. The Na spectrum is cropped in amplitude, the peak amplitude in the above units is 58.

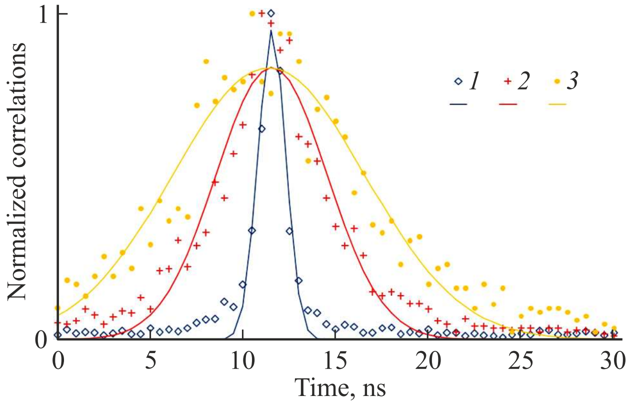

Figure 3. Example of normalised photon correlation spectra (symbols) and their Gaussian approximations (solid curves) obtained at SL of 3 M NaCl solution with the use of light filters: „blue“, 250–400 nm (*1*); „red“, 650–800 nm (*2*); „yellow“, 585–595 nm (*3*). *W* is equal 1.7, 7.1, 12.2 ns respectively.

Fig. 3 shows photon autocorrelation spectra and their approximation by Gaussian curves on the example of a 3 M NaCl solution obtained with three different light filters cutting the continuum and spectral intervals of different widths, including the Na line, from the SL spectrum.

In [8] the duration of the continuum flashes was determined as ~ 1.8 ns and was the same for all wavelengths. The SL continuum is formed by processes caused by adiabatic heating of the bubble, with the thermal mechanism not being the direct source of the luminescence. Heating leads to ionisation of particles, dissociation of molecules, and formation of various excited states. In [14,15] the mechanism of luminescence of the excited, including unbound states of molecules, at high-temperature chemical processes in bubbles is proposed. The duration of these processes, based on the duration of the flash, is less than 1 ns.

The duration of the luminescence flashes of metal atoms at SL according to our measurements is of the order of ~ 10 ns, depending on the wavelength according to the results [16] and, as shown below, on the excitation energy and atomic mass of the metal. The gas environment of the bubble at times close to the collapse peak consists mostly of inert gas, which forms the excimers „alkali metal–inert gas“ responsible for the blue satellite [13]. In [13] the mechanisms of chemical excitation of metals with participation of radicals are also considered. Two mechanisms for the appearance of free neutral atoms of alkali metals inside the bubble —microdroplet and layer [17]. Judging by the duration of the flash, the times of these processes are also ~ 10 ns.

Note that the natural lifetime of the excited states of free alkali metal atoms (17–32 ns) has little relation to the duration of their flashes at SL. As measurements have shown, the delay of the Na flash after the continuum flash is a fraction of ns, whereas the delay of the Ce^{3+} flash after the continuum is ~ 31 ns, which coincides with the lifetime of its excited state [16]. It is possible that for alkali metal atoms there is strong non-radiative quenching of excited states in contrast to Ce^{3+}.

Effects of atomic mass and excitation energy

Figure 4 shows the duration of the flashing of metals at SL of 3 and 1 M solutions of their chlorides as a function of the atomic mass of the metal. When the atomic mass is increased by a factor of ~ 12, the *W* increases approximately twofold; this effect is more pronounced for 1 M solutions. In [18] it was shown that the brightness of the OH radical band (310 nm) at SL is proportional to the square root of the reduced mass of the emitter (OH radical molecules) and quencher (atoms of different noble gases) particles. A similar mechanism of nonradiative quenching of excited states may be relevant in this case and will affect not so much the brightness as the fluorescence lifetime (emitters — metal atoms of different masses, quenchers — argon atoms).

Fig. 5 shows the duration of the metal flash at SL as a function of the excitation energy of the E_{ex}. When increasing E_{ex} by 0.5 eV, the *W* decreased by about half. This can be explained within the framework of the dynamical model of metal line shape [9] — the residence time of the bubble at lower temperature (in a less energetic state) is longer, and thus a metal with low excitation energy has the ability to be emitted for a longer period of time. The Na flash spans the time continuum flash, the middles of these flashes are almost synchronous [16], and we believe this is true for other alkali metals. Developing the idea, we can plot the time dependence of E_{ex} by reversing the axes in drawing 5 and plotting the values of *W* around the zero of the time axis (taken as the collapse peak), which is shown in drawing 6 for SL from 1 and 3 M salt solutions. In figure *W* are averaged and corrected for the value of W_0. A Gaussian approximation of the point distribution is performed, which gives the expected course of the

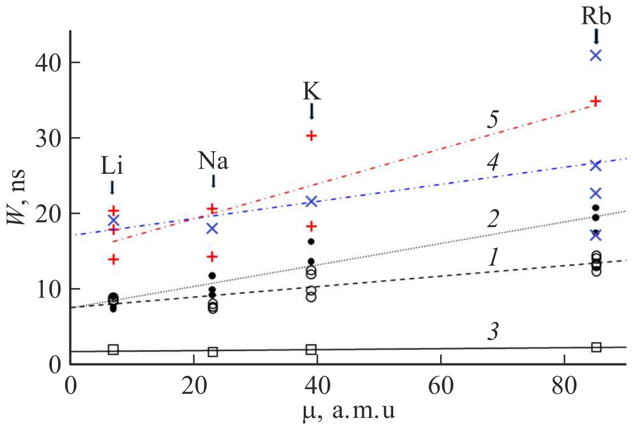

Figure 4. The duration of the flash of metal atoms in related to their atomic mass in SL solutions of concentration 3 M (*1*) and 1 M (*2*). The duration of the continuum flash in the same solutions (*3*). The flash duration of metal atoms in solutions of 2 mM concentration with addition of 2 mM SDS (*4*). Broadband filters were used, $\Delta\lambda \approx 200$ nm. The flash duration of alkali metal 3 M solutions in the region of their spectral lines in a narrow spectral range $\Delta\lambda \approx 5$ nm (*5*). A linear approximation has been performed.

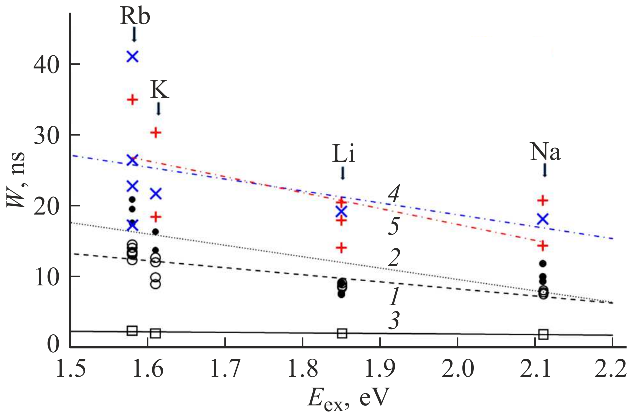

Figure 5. The flash duration of metal atoms in related to their excitation energy at SL of solutions of concentration 3 M (*1*) and 1 M (*2*). The duration of the continuum flash in the same solutions (*3*). The flash duration of metal atoms in solutions of 2 mM concentration with 2 mM SDS (*4*). Broadband filters, $\Delta\lambda \sim 200$ nm were used. The flash duration of the metal atoms of 3 M solutions in the region of their spectral lines in the narrow spectral range is $\Delta\lambda \sim 5$ nm (*5*). The continuum is conventionally labelled on the same E_{ex} as the line of the metal atom. A linear approximation has been performed.

emitter luminosity of different energies during collapse. Confusingly, the asymptote is at 1.6 eV (this corresponds to $T \sim 18000$ K), but there is no thermodynamic equilibrium in the bubble close to the moment of collapse. As known from analytical spectroscopy, the suitable temperature for alkali metal emission is much lower and is 2000–2500 K [19], 1700–1900 K [20,21], i.e. of the order of 0.2 eV.

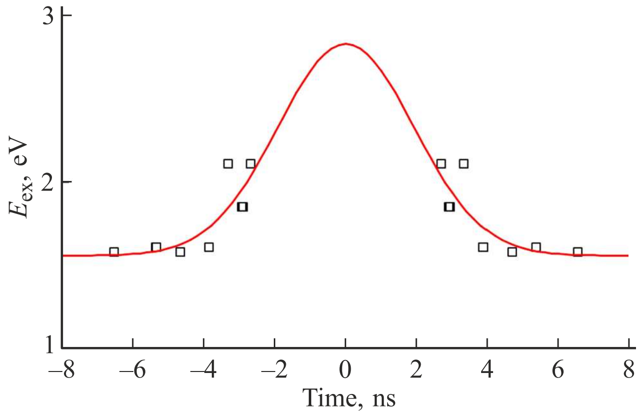

Figure 6. Simulation of the energy change in the collapsing bubble at SL of 1 M and 3 M solutions by flash durations and excitation energies of alkali metals. The collapse is assumed to be symmetric on the time scale. The durations are corrected by W_0 and correspond to the time-scale distances between points symmetrical about the collapse peak.

The duration of the flash near the centre of the Na line

As shown in Fig. 4, 5 (curves *5*), the W of the metal in the narrow spectral range near the line centre is about twice as large as that in the broad spectral range. This means that the narrow component, which is only possible at low medium densities, is emitted for a longer duration than the density-widened line component, which is consistent with the dynamic line shape model [9]. This result was previously detected for NaCl solution in [16] and confirmed for LiCl, KCl, and RbCl solutions in the present study.

Figure 7 shows the results of W measurements for a range of NaCl concentrations in narrow spectral intervals using a monochromator ($\Delta\lambda \sim 5$ nm) along the Na spectral band where the brightness was sufficient for correlation measurements. It can be seen that the W immediately near the centre of the line is larger than in its vicinity.

Note that the spread of W values when measured near the centres of the metal spectral lines is quite wide, as can be seen in figs. 4 and 5, as well as for similar data in figs. 3, *a* and 3, *b* in [8]. Since the measurements of the W continuum [8], as well as the flash durations of Ce^{3+} [6], give fairly stable results, this variation in the data cannot be attributed to noise introduced by the correlation counter. The reason should be sought, as we believe, in the mechanism and localisation of metal emission in the bubble cloud. The „sonoluminescent“ and „sonochemical“ zones [22,23] are distinguished. Continuum emission originates from the sonoluminescent zone, where the bubbles experience nearly spherical and nearly simultaneous collapse. Metal atom emission occurs in the sonochemical zones from bubbles undergoing surface deformation as they fragment and form streamers [24]. The collapse of „sonochemical“

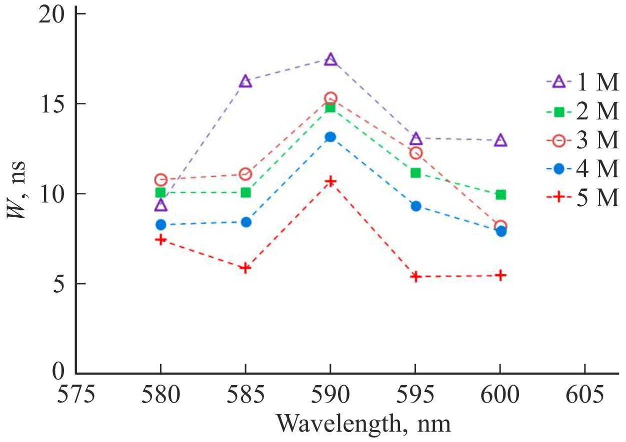

Figure 7. The duration of the Na flash in solutions of 1–5 M NaCl measured in narrow spectral intervals for five wavelengths.

bubbles is chaotic in time and intensity, which, in our opinion, is the reason for the spread of W values of alkali metals.

Concentration effect

In 3 M solutions of alkali metal chlorides, the W was smaller by about a quarter than in 1 M solutions (Figs. 4, 5). A more detailed study of NaCl solution over a range of concentrations in spectral intervals of different widths showed that the duration of the Na flash decreases as the concentration of the solution (Fig. 8) increases. For the measurements we used an MDR monochromator (588–593 nm), a narrowband BP589 filter (585–596 nm), broadband filters (550–800 nm) and for comparison a filter passing only the SL continuum (250–400 nm). It was previously shown that the W of the continuum is independent of wavelength over the entire available range of the spectrum [8]. Here we have obtained that the $W \sim 1.8$ ns (without correction for the intrinsic W_0) is also independent of salt concentration.

We believe that the decrease in the W of the metal as the concentration (Fig. 8) increases is due to a decrease in the average size of the cavitation bubbles and, as a result, a narrowing of the time interval of their collapse. Bubble size, according to [25], decreases significantly with concentration. The data [25] obtained in NaCl solution for an ultrasonic frequency of 355 kHz are shown as a graph in Fig. 9. The bubble size at 20 kHz is larger than at 355 kHz [22], but obviously the size-concentration dependence must remain. To estimate the duration of the bubble compression phase, we used Rayleigh's solution for the collapse time of an empty spherical cavern of radius R in a liquid of density ρ under an applied pressure P: $t_0 = 0.915R(\rho/P)^{1/2}$. The values of R are taken from [25], our measured values of ρ for different concentrations increased from 1000 to 1180 kg/m^3, the value of $P = 10^5$ Pa is adopted. The calculation results are shown in Fig. 9. Since t_0 is directly proportional to R, its graph practically repeats the bubble size graph. Since the flash occupies only a part of the compression phase, the Rayleigh collapse time reasonably agrees with the measured W of the metals.

A significant increase in the continuum brightness and a drop in the brightness of the Na line at high NaCl concentrations cause broadband filters that pass a significant fraction of the continuum to show a decrease in W to almost 2 ns. This is not due to the physics of the process, but to the observational conditions, and we believe it is explained by a decrease in the fraction of the Na line brightness in the filter transmission region. The fraction s of the Na line brightness in the bands cut out by the filters was calculated from the SL spectra of NaCl solutions of the corresponding concentration, the SL spectrum containing only the continuum, and the transmission spectra of the filters (Fig. 10).

Let us compare our results with those of [19] on the collapse of a single bubble generated by a laser pulse

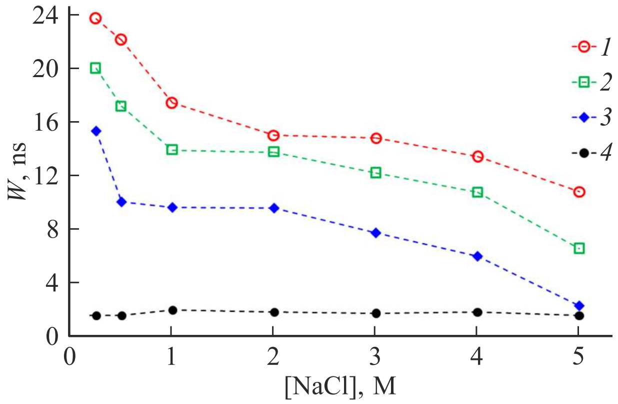

Figure 8. The dependence of Na flash duration on NaCl concentration in spectral intervals including the Na line of different widths: $\lambda \sim 5$ (*1*), 11 (*2*), 200 nm (*3*). The duration of continuum flashes (*4*) is given for comparison.

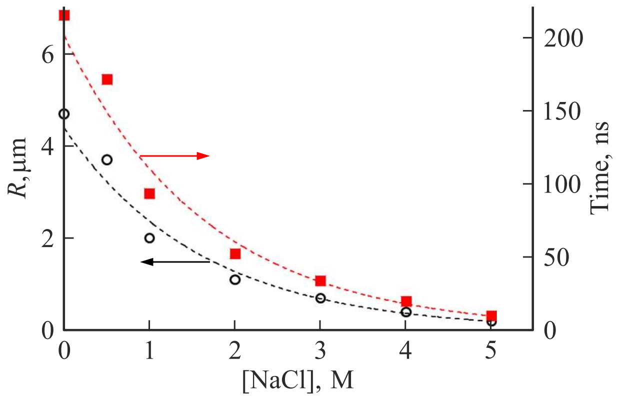

Figure 9. Bubble size in NaCl solutions of different concentrations from data [25] (circles) and calculated Rayleigh bubble collapse time (squares). An exponential approximation was performed.

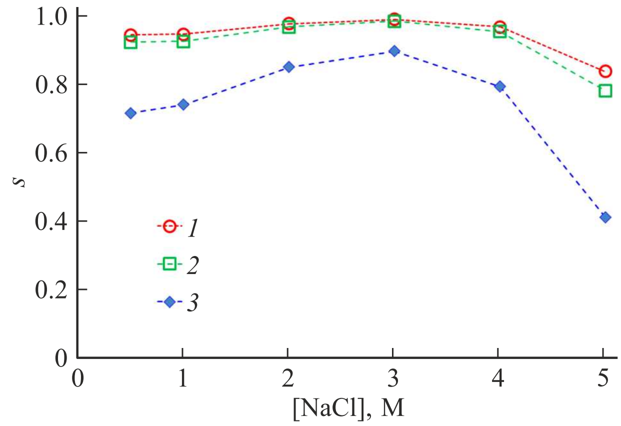

Figure 10. The fraction s of the Na line brightness in the cutoff spectral range for the cases *1, 2, 3* shown in Fig. 8.

in NaCl solutions of different concentrations, where the Na flash duration decreased from ~ 135 to ~ 30 ns as the concentration was increased from 0.2 to 6 M. The authors [19] attributed this to two mutually complementary effects that narrow the flash on both sides of the time scale. Resonance trapping of radiation by Na atoms leads to an increase in the effective fluorescence lifetime τ_0 of free Na atoms and increases with higher concentration, pushing the onset of Na luminescence away from the time taken as the onset of excitation (100 ns before the collapse peak), on one side. Non-radiative quenching of excited states reduces τ_0 in a dense medium near the collapse peak, which shortens the Na flash time, on the other hand.

In our case at SL, we observe a similar trend of decreasing Na flash duration as the solute concentration increases. We suggest another explanation. In our case, the dependence of the mean bubble size on concentration makes a significant contribution. Resonance trapping of the emission should itself lead to an increase in flash duration. Although resonance trapping is estimated in [19] as significant, we believe that it is suppressed by non-radiative quenching processes. This conclusion is supported by the near-zero measured delay of the Na flash after the continuum [16] flash. The synchronicity of the Na and continuum flashes in the SL case contrasts with the distinct ordering of the flashes during the collapse of the bubble generated by the laser pulse. The reason for this difference is unclear and requires further investigation.

When analysing the Na flash duration in bubbles, the following processes should be considered: (1) conditions under which the existence of free Na atoms is possible, (2) the switching on and off of chemical reactions leading to the excitation of Na atoms, (3) intensive non-radiative quenching of excited states in a dense medium, and (4) the nonsphericity of Na-emitting bubbles, which transforms the collapse from a high-energy „short“ event into a more extended process affecting different regions of the bubble.

Surfactant effect

The luminescence flash durations of metal atoms obtained using broadband filters in solutions of 2 mM SDS (for Na), 2 mM SDS+2 mM MCl (M=Li, K, Rb), are approximately twice as long as the flash durations in salt solutions of higher concentrations without surfactant (Figs. 4, 5).

The addition of surfactant allows spectral lines of metals to be observed at low concentrations: the Na line is observed in 0.1 mM SDS solution, whereas in NaCl — solution starting at 0.1 M. The enhancement effect is understandable because the surfactant concentrates the metal ions at the bubble−solution boundary. The presence of any type of surfactant (cationic, anionic, nonionic, zwitterionic) in the solution of alkali metal salt results in enhancement of its spectral lines at SL [26] and narrowing of their spectral profile [27]. The narrowing of the line represents a decrease in the density of the emitting medium, which is assumed to correspond to longer time intervals encompassing the moment of bubble collapse.

Note the trend in SL, including without added surfactant: the narrower the spectral line of the metal, the longer the duration of the flash. The relationship of these two factors to the average bubble size is ambiguous. Thus, the bubble size at low salt concentrations increases [25] and decreases [28,29] when surfactant is added.

The shape of the line can change under different experimental conditions, particularly when the salt concentration is changed, but here too the observations are contradictory. Both narrowing, broadening, and unchanged shape of the Na line were observed as the concentration increased. Thus, in [30], the width of the Na line in 1 M NaCl solution is noticeably larger than in 5 M solution, the asymmetry of the line is more pronounced in the spectrum of 1 M solution, and the doublet of the line is clearly expressed in the spectrum of 5 M solution. In [27], we observed a constant shape and width of the Na and K lines for 0.5 and 5 M solutions of NaCl and KCl. In [31], the shape of the Na line and its intensity were dependent on the ultrasound power and changed with the addition of ethanol. In the single-bubble SL spectrum of 0.1 mM SDS solution, only the narrow components of the Na doublet were visible and the contribution of the broad component was imperceptible, whereas in the multi-bubble SL of 4 M NaCl solution, the Na doublet was visible against a prominent background of a broadened [12] line.

Conclusion

Using the time-correlated single photon counting method, the duration of the flashes of the atomic lines of alkali metals Na (589 nm), Li (671 nm), K (767 nm), Rb (780 nm) during multibubble SL of aqueous metal chloride solutions was measured to be 5−40 ns.

The duration of the metal line flashes increased by a factor of two, following a 12-fold increase in the atom mass and/or a one third decrease in the excitation energy of the metal atom. The mass effect is related to quenching of the excited states, while the excitation energy effect is associated — with the energy dynamics in the bubble at its collapse. Based on the data obtained, a model of the course of energy in the bubble has been built.

The duration of flashes in the narrow spectral interval including the spectral line is approximately twice as long as in the wide interval. This is consistent with our proposed line shape model.

The duration of flashes in 1 M solutions of Na, K, and Rb salts is longer than in 3 M solutions by about a quarter. In series of NaCl concentrations, the flash duration of the Na line decreased monotonically, indicating that the average bubble size decreased as the salt concentration increased.

The flash duration of metal atoms in solutions of low concentration increased with the addition of surfactants. Surface-active substances attract metal ions to the surface of bubbles and increase the brightness of metal lines at SL by 2−3 orders, lower the surface tension, increase the viscosity of the solution, increasing the time at which the conditions in the bubbles are suitable for emission of metal lines.

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Conflict of interest

The authors declare that they have no conflict of interest.

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