

Composition and its evolution inside a sonoluminescing bubble by line spectra

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The line emissions of OH^* , Na, Na-Ar^{*}, and Ar are observed in stable and bright single bubble sonoluminescence (SBSL), which shows that the composition of the bubble consists of at least three parts: the vapor, droplets of the host liquid, and the gas dissolved in the host liquid. The observation of line emissions in SBSL demonstrates that it shares exactly the same spectra with multibubble sonoluminescences (MBSL). The experiments indicate that noble gas plays an important role in all line emissions of both SBSL and MBSL. The time resolved spectra of SBSL show that there is significant mass exchange between the inside and outside of the bubble. The time scale of the mass exchange ranges from less than $\frac{1}{10}$ s to tens of seconds. The SBSL in sulfuric acid supports the argon rectification theory. Because stable SBSL in sulfuric acid can just be achieved within a narrow parameter range, only weak rectification is observed in our experiments.

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I. INTRODUCTION

The light emission from cavitation bubbles driven by ultrasound was observed 70 years ago, which is now called multibubble sonoluminescence (MBSL) to be distinguished from the single bubble sonoluminescence (SBSL) [1,2] discovered by Gaitan in 1989. The differences between MBSL and SBSL are not only in the number of the luminescing bubbles, but also in the lifetime of the bubbles. The composition of an SBSL bubble may be different from that of an MBSL bubble. An argon rectification hypothesis has been proposed in SBSL due to its pulsative and periodical oscillation [3,4] and validated by experiments [5] according to the difference of the response time to a sudden change of the driving pressure between an air bubble and an argon bubble. The so-called argon rectification hypothesis [3] infers that oxygen and nitrogen gases inside an air bubble are converted into chemically reactive species that are quickly and irreversibly transferred to the host liquid, while the nonreactive argon gas will remain in the bubble. The MBSL bubbles probably do not survive for enough cycles to rectify the argon [5]. Hence, for the air-water system, the gas trapped in the SBSL bubble is different from that in the MBSL bubbles. Besides, many theoretical works [3,6–9] predicted the water vapor would be trapped in the bubble because the final phase of collapse is so fast that vapor could not readily diffuse out of the bubble. Unfortunately there is still a lack of the direct experimental investigation about what is really inside an SBSL bubble.

Another remarkable difference between MBSL and SBSL is in their spectra. The MBSL spectrum is a broadband spectrum with some lines, such as the OH^* radical line (310 nm) [10] in water and the sodium lines (589 nm) [11,12] if the host liquid contains the sodium salt, while the SBSL bubble spectrum was presumed to be a featureless continuum in early experiments [13] until Young *et al.* reported their observation of the OH^* radical line emissions in extremely dim SBSL [14]. Recently argon atomic lines were also observed in moving SBSL from concentrated aqueous sulfuric acid

(SA) [15,16]. The strength of the luminescence was not as low as that reported in Ref. [14] though the bubble was not steadily levitated at a fixed position. As is well known, the line emissions reflect the characteristic of the illuminant, and one usually detects the composition of a sample by measuring its line spectra, which is simple but very effective. Therefore the discovery of the line emissions of the SBSL spectra [14–16] opens a new prospective in understanding the composition of the luminescing bubble. A reliable measurement of the composition of the bubble requires the luminescing bubble to be stably levitated during the exposure period of spectrometer, because the movement of the bubble may cause not only measuring errors in the spectrum but also induce extra significant mass exchange between the inside and outside of the bubble. Because of this we desire to investigate the composition and its evolution with time by measuring a stable and bright SBSL spectra, rather than a moving and extremely dim SBSL spectra.

The paper is organized as follows. In Sec. II we introduce the experimental technique to form stably levitated sonoluminescence. In Sec. III we show the observations of the line spectra which reflect the composition of a luminescing bubble. In Sec. IV we study the evolution of the line spectra. Finally, in Sec. V we summarize the main conclusions together with some further discussions.

II. EXPERIMENTAL APPARATUS AND TECHNIQUE

The main experimental apparatus of SBSL consisted of a spherical acoustic resonator (see Fig. 1) and a spectral acquisition system. The acoustic resonator, a 100-ml glass flask with a quartz window, was driven by ultrasound at the resonant frequency of about 25 kHz. The apparatus of MBSL mentioned in this paper included an ultrasonic generator at frequency 20 kHz (Ultrasonic VCX750) and a stainless steel box with a quartz window. An Acton Research SP-300i imaging spectrometer with a Princeton Instruments liquid nitrogen cooled charge-coupled-device detector was used to acquire spectra.

The host liquids used here were aqueous SA (the concentration was 85 wt % if not noted otherwise) and deionized distilled water. Some alkali metal salts, such as sodium chlo-

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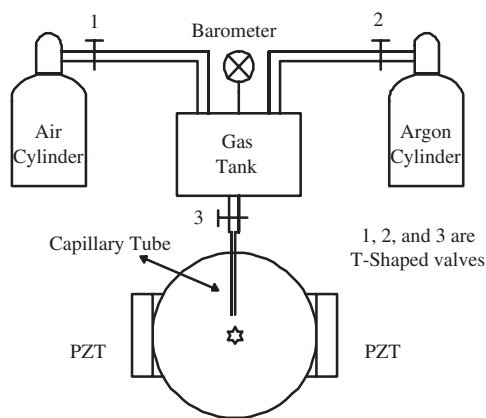


FIG. 1. The cell and the gas injecting part for SBSL. The resonant frequencies of the cell are about 26.6 kHz for SA and about 25.6 kHz for water.

ride, sodium sulfate, etc., were dissolved into the host liquid in parts of the experiments. All of the SA used in the experiments was degassed by keeping it in vacuum for at least 10 days, and then regassed with particular gas, such as argon or air, for 2 days under pressure 20×133.3 Pa in the SBSL experiments. During the whole process of gas preparation, a magnetic rotor covered with teflon was used to beat the solutions to speed up the degassing and regassing processes. For water, both degassing and regassing processes lasted 2 days, and the regassing pressure was 50×133.3 Pa instead of 20×133.3 Pa. However, the regassing pressure was about 500×133.3 Pa for the MBSL host liquid. The effective driving pressure P_a of the sound field was determined by fitting the Mie scattering data to the bubble radius $R(t)$ [17] (this effective P_a could differ substantially from the value extrapolated from hydrophone measurements in the noncavitating fluid).

In the experiments, the bubble was seeded by injecting gas through a glass capillary tube fixed at the upper center of the resonator. The injected gas was from a gas tank which connected to two cylinders filled with different gases, air and argon for example. A barometer controlled partial pressures (see Fig. 1). In most of our experiments the injected gas was simplex air or one kind of noble gas, but in a special group of experiments the mixed gases of air and argon were used.

All spectra were acquired by spectrometer with the 0.200-mm slot width and calibrated for the absorption of the host liquid and the resonator wall against standard lamps. The host liquids were kept at room temperature, 18 °C, if not noted otherwise.

The spectra of the moving SBSL in concentrated SA was reported with clear argon atomic line emissions [15,16]. In our experiment, we endeavored to improve the stability and finally achieved stably levitated SBSL in concentrated SA. A stably levitated bubble in SA was photographed with an exposure time of $\frac{1}{30}$ s (see Fig. 2). The clear profile of the bubble's image proves its stability in SA. Furthermore, we also observed the same line emissions of argon as that reported in Ref. [15]. The most important factors to obtain a stably levitated bubble were regassing the host liquid at a low pressure, say less than 50×133.3 Pa for both air and

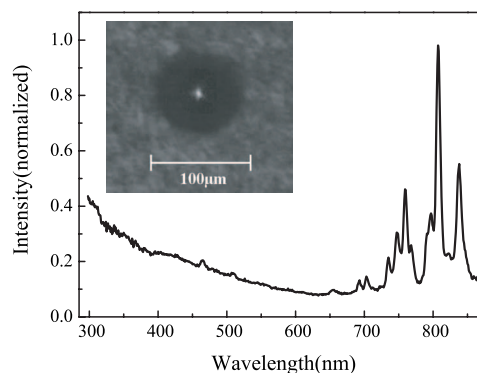


FIG. 2. (Color online) Line spectrum of a stable and bright SBSL. The inset is a microscopic picture of the bubble. The time of exposure is $\frac{1}{30}$ s. A central white point in the picture is SBSL light. The driving parameters are $f=26.6$ kHz and $P_a=1.22$ bars.

argon after strictly degassing, and carefully seeding the gas bubble.

III. LINE EMISSIONS AND COMPOSITION

Because the spectra of well-known SBSL [2] in water are always a featureless continuum, little research has been conducted in studying the composition of the SBSL bubble by spectra. However, after systematical study about the argon-water SBSL, we observed various new line emissions. From these lines the composition inside the bubble can be deduced.

A. New line emissions of SBSL

First, we observed the line emission of OH^* radical (310 nm) from argon-water SBSL. At the beginning, when we set the driving pressure at about 1.11 bars, we got a spectrum with a very high OH^* radical peak (see dashed-line in Fig. 3). The OH^* radical peak became lower and lower as we increased the acoustic driving pressure step by step, and disappeared when the driving pressure reached 1.32 bars. All the light emissions were bright enough to be observed by the

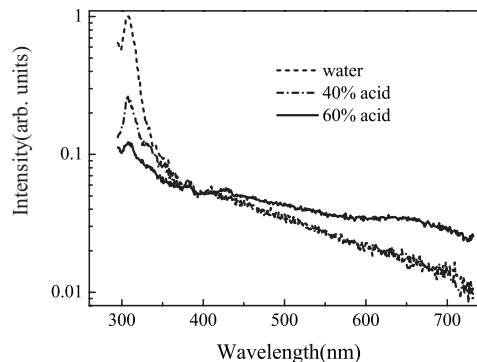


FIG. 3. Line spectra of stable and bright SBSL. The host liquids were water, 40 and 60 wt % SA (aq.). The concentration of the argon in the host liquids was about 50×133.3 Pa. The driving parameters are $f=25.6$ kHz for water and about 26.0 kHz for SAs, and $P_a=1.11$ bars, respectively.

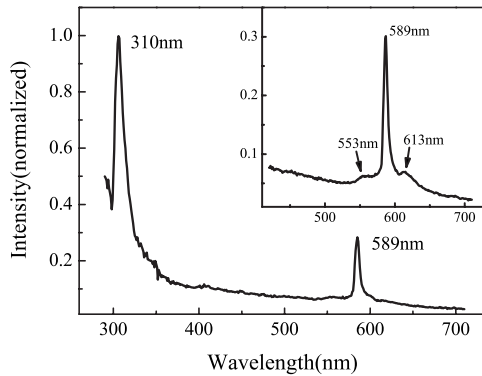


FIG. 4. Sodium line emissions in stable and bright SBSL from the deionized distilled water dissolved with sodium sulfate. The driving parameters are $f=25.6$ kHz and $P_a=1.11$ bars.

naked eye, and the typical exposure time was about 2–5 min other than the 20-min exposure time for the dimmest bubble. Hence OH^* line emissions at 310 nm were observed in the stable and bright SBSL. Here it is important to point out that the necessary condition for the OH^* radical line emission is that the host liquid must be dissolved with noble gas under the pressure $\geq 20 \times 133.3$ Pa. We found that the OH^* radical emission could be observed when the gas pressure ranged from 20×133.3 Pa to 150×133.3 Pa.

Second, we observed both OH^* radical and Na atomic line emissions in the SBSL from water dissolved with 50×133.3 -Pa argon gas and about 3 mol/l sodium sulfate (see Fig. 4). We found that the peaks decreased with higher driving pressure. It is interesting that there were two small peaks nearby the sodium lines symmetrically. The left one at about 553 nm was reported in MBSL and called blue satellite spectrum [18]. The right one, named red satellite spectrum, was first observed here. Both of them should come from electronically excited Na-Ar^* molecular [18].

We remind the readers that all the line emissions of SBSL reported here will disappear if the host liquid is not dissolved with any noble gas. This fact is also true in the corresponding MBSL experiments. Figure 5 shows a spectrum comparison of MBSLs in water dissolved with and without argon gas. The line at 310 nm disappeared when the water did not con-

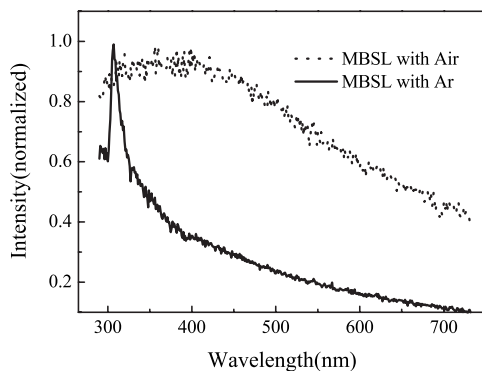


FIG. 5. Spectrum comparison between MBSLs in water dissolved with argon (solid curve) and air (dot curve). The frequency is at $f=20.0$ kHz and the output power of VCX750 is set in 30% $\times 750$ W.

tain argon. In fact, almost all line emissions of MBSL in previous work [10,11] were observed in the host liquids saturated with noble gas. Although in Ref. [13] some line emissions had been observed in water under air sparge, one of the authors believed that the argon in air played an important role in line emissions [19]. Surprisingly, even if noble gas is dissolved into the host liquid, the line emission of MBSL could disappear [20] just like that of SBSL in water [14] and SA [15] did with the increase of the driving pressure.

Based on the above observations we can draw the conclusion that stable and bright SBSL can share exactly the same spectra with MBSL. All line emissions reported here were observed in the bubble contained argon gas. We conducted similar experiments using krypton instead of argon, and the results were completely consistent with those using argon. Therefore it is of significance to point out that noble gas is a key factor to make the line emission appear in sonoluminescence. In addition, the driving pressure is another important factor to determine the relative intensity of all the emitted lines.

B. Composition of a luminescing bubble

Vapor of the host liquid. Since we observed numerous line emissions of SBSL which used to be only observed in MBSL, we can analyze the matter composition inside the bubble by spectra.

Although the OH^* radical line emission could be from either the vapor or droplets of water, we attribute it to the water vapor. Figures 1 and 3 show the intensity of OH^* radical line emissions in solutions of SA and water at different concentrations. It is understandable that the intensity of OH^* radical line emission decreases with the percentage of water in the host liquid. As is well known, the water molecules are bound by SA in concentric SA due to its significant hygroscopicity. For example, in 85-wt % SA solution there is almost no water vapor inside the bubble. Obviously there are still water molecules in concentrated SA as part of the solution. Furthermore, the number of water molecule is almost as large as that of SA in 85-wt % SA because their molecular weights are quite different. As a part of the concentrated SA, droplets contain a large amount of water molecules. Therefore the fact that there is no OH^* radical line emission observed in concentrated SA (Fig. 2) supports the hypothesis that the line emission comes from the gas phase instead of the liquid phase of water, that is, from its vapor instead of droplets. It also agrees with the observations of OH^* radical line emissions in different SA solutions (Fig. 3). As the concentration of SA increases, the intensity of the line emission decreases.

Droplets of the host liquid. Further experiments showed that there are also the droplets of the host liquid inside the bubble, which was proposed in MBSL [18]. One clue is from the sodium atomic line emissions of SBSL in the argon-SA system, mixed with sodium sulfate powder. Normally, due to the very low dissolvability of sodium sulfate in concentrated SA solution, sodium lines cannot be observed in concentrated SA, no matter how much sodium sulfate was mixed into the concentrated SA solution. Interestingly, when so-

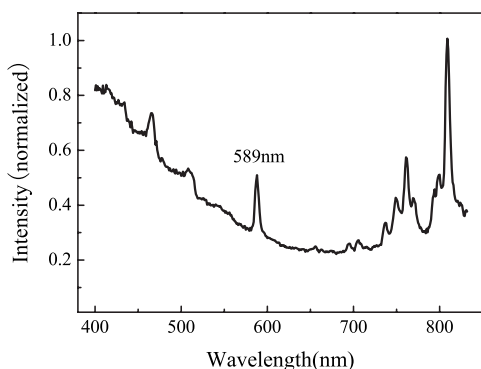


FIG. 6. Sodium line emissions in stable and bright SBSL from the slurry of 85-wt % SA and powder of the sodium sulfate. The driving parameters are $f=26.5$ kHz and $P_a=1.32$ bars.

dium sulfate powder was mixed into the concentrated SA solution, the sodium atomic line emission at 589 nm could occasionally be observed (see Fig. 6) in their slurry, although there was no line at 589 nm most of the time. It is easy to conclude that some powder was involved into the bubble in a droplet. Further evidence for the existence of the droplets is the emission of the electronically excited Na-Ar^* molecule, blue and red satellites (see Fig. 4). The excited molecule needs the excitation of Na in the gas phase [18], which requires the mechanical addition of alkali metal salts to the collapsing bubble since the vapor pressure of alkali metal salts are too small for their evaporation to be plausible [for example, $P_{\text{vap}}(\text{NaCl})=1$ mm Hg at $T=1200$ K [18]]. A convincing explanation is that the outside host liquid invaded the bubble as droplets.

Gas dissolved in the host liquid. As shown in Fig. 2, argon lines are observed in SBSL from concentrated SA dissolved with argon. If other noble gases are dissolved instead of argon, their lines can also be observed. So it is straightforward that the noble gas dissolved in the host liquid has invaded into the luminescing bubble. In fact, in the next section we will discuss in detail what kind of gas is in the bubble and the results will show that any gas dissolved in the host liquid will invade into the bubble.

IV. MASS EXCHANGE ACROSS THE BUBBLE WALL

The observation of the OH^* radical and alkali metal atomic lines has not only indicated the composition inside the bubble, but also shown the host liquid invading the bubble. It is deducible that the composition of the bubble will approach a steady state which may differ from the initial state. The initial matter trapped in the bubble will change by exchanging between the inside and outside of the bubble during the pulsation. Since noble gas is a key factor of all the line emissions, further experiments were conducted to study the mass exchange across the bubble wall together with the argon rectification in SBSL.

A. Time scale of the exchanges

To study the mass exchange, we set the spectrometer for successional shooting without intervals. The exposure time

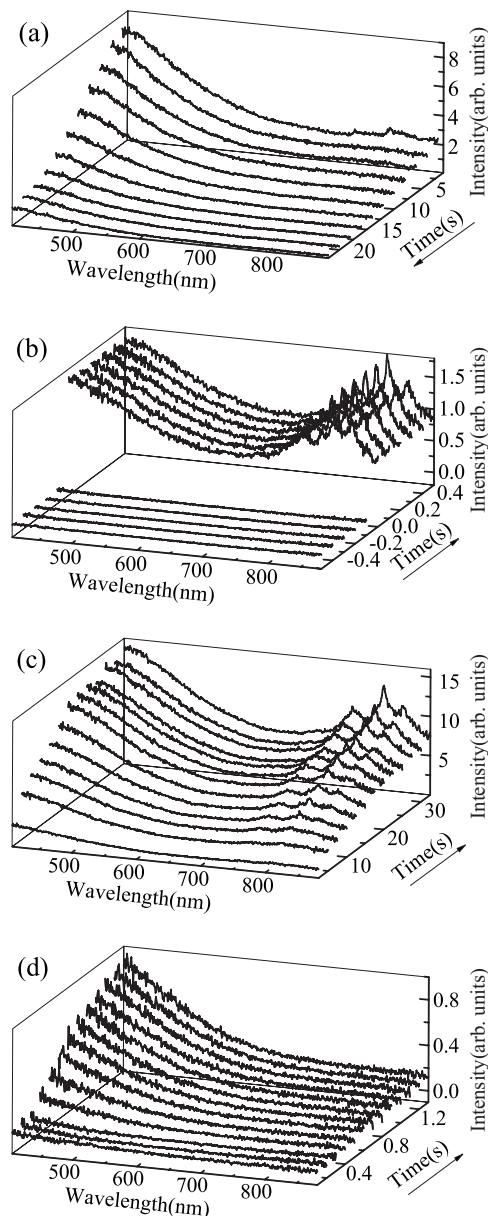


FIG. 7. The evolution of the spectra of stable and bright SBSL in SA. (a) An injected argon bubble in SA dissolved with 20×133.3 Pa air, (b) an argon bubble in SA with 20×133.3 Pa argon, (c) an air bubble in SA with 20×133.3 Pa argon, and (d) an air bubble in SA with 20×133.3 Pa air. The bubbles are seeded at the time 0. The driving parameters are the same: $f=26.6$ kHz and $P_a=1.32$ bars.

was set ranging from 0.1 to 2 s for each spectrum. The experiment included measurements of time-resolved spectra of SBSLs in cases (i) Ar-in-air, an injected pure argon bubble in SA dissolved with 20×133.3 Pa air, (ii) Ar-in-Ar, (iii) air-in-Ar, and (iv) air-in-air. Figure 7(a) shows a spectrum evolution of a bright-to-dark sonoluminescence from an injected pure argon bubble in SA with 20×133.3 Pa air. As mentioned in Ref. [16], the optimized gas for sonoluminescence is noble gas since it is neither condensable nor reactable. Therefore the bright-to-dark luminescing shows that the initial injected argon inside the bubble transfers to the outside

gradually. This observation can be understood in the theoretical framework of gas diffusion. If we use

$$\frac{dM_{Ar}}{dt} = 4\pi R^2 D_{Ar} \left. \frac{\partial C_{Ar}}{\partial r} \right|_{r=R} \quad (1)$$

to express the change rate of argon inside the bubble [3,21], where $\left. \frac{\partial C_{Ar}}{\partial r} \right|_{r=R}$ denotes the gradient of the concentration of the argon on the bubble wall, D_{Ar} is the diffusion coefficient of argon and R is the bubble radius, respectively. Considering the argon rectification, we need another formula,

$$\frac{dM_{N_2}}{dt} = 4\pi R^2 D_{N_2} \left. \frac{\partial C_{N_2}}{\partial r} \right|_{r=R} - AM_{N_2} e^{-(T^*/T)}, \quad (2)$$

to describe the change rate of other active gases in air, mainly nitrogen [3], where the subscript N_2 denotes the nitrogen gas, A is a coefficient related to the time scale of the chemical reaction which follows an Arrhenius law, and T and T^* are the temperature and the activation temperature, respectively. For an injected pure argon bubble $\frac{dM_{Ar}}{dt} < 0$ due to $\left. \frac{\partial C_{Ar}}{\partial r} \right|_{r=R} < 0$, so that the concentration of argon inside the bubble will decrease. The air will diffuse into the bubble due to $\left. \frac{\partial C_{N_2}}{\partial r} \right|_{r=R} > 0$. According to the argon rectification theory [3–5,22], the dissolved air will be rectified, which lifts the concentration of argon in the bubble. As a result of the diffusion and rectification, the composition will arrive at a new equilibrium state with the surrounding host liquid finally. The transition time, the time scale of the mass exchange, appeared to be at the order of ten seconds [see Fig. 7(a)]. If the host liquid was dissolved with argon too (Ar-in-Ar), the evolution of the spectrum would relate to the concentration of argon outside the bubble. We found that the spectra kept the same exactly as soon as the bubble luminesced, if the dissolved argon $> 20 \times 133.3$ Pa. The detailed transition process was not observable because of the limitation of the shortest exposure time of the spectrometer, 0.1 s [see Fig. 7(b)]. Contrary to the case of Ar-in-air, a dark-to-bright sonoluminescence was observed from an injected air bubble in SA with argon [see Fig. 7(c)]. The transition time was also at the order of 10 s. For this case $\left. \frac{\partial C_{Ar}}{\partial r} \right|_{r=R} > 0$ so that the argon gas will diffuse into the bubble at the beginning. Furthermore, the residual air in host liquid will diffuse into the bubble and be rectified, speeding up the increase of the concentration of argon. If the inside argon concentration were larger than the outside, $\left. \frac{\partial C_{Ar}}{\partial r} \right|_{r=R} < 0$, $\frac{dM_{Ar}}{dt}$ would become negative. So the composition inside the bubble arrives at an equilibrium state, and the argon concentration becomes constant. Also, another dark-to-bright sonoluminescence was recorded in the case of air-in-air [see Fig. 7(d)], which is a typical event of the argon rectification [3–5,22]. A gradual increase of SBSL intensity supports the statement that the gas inside the bubble is rectified and the argon concentration increases. At the same time, we notice that there were no argon atomic lines observed in Fig. 7(d). The time scale of this transition, however, was only about 1 s, about ten times less than the gas diffusion time scale.

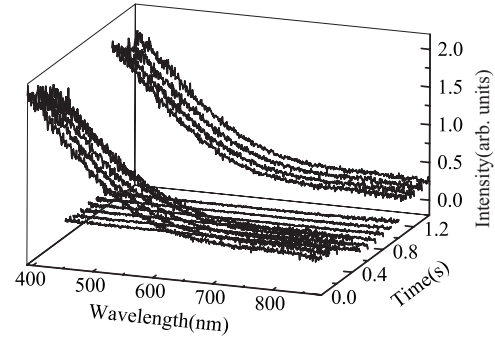


FIG. 8. The spectra from a luminescing air bubble (in 85-wt % SA) driven by jumping pressure. The pressure was turned off from 0.5 to 1.0 s. The air dissolved in the solution is 20×133.3 Pa. The driving parameters are $f=26.6$ kHz and $P_a=1.32$ bars.

B. Argon rectification

To further study the argon rectification in our air-SA system, we conducted another SBSL driven by a jumping pressure, similar to that used in Ref. [5]. We levitated a luminescing bubble and maintained it for several minutes. Then we instantly shut down the driving pressure, and recovered it half a second later (see Fig. 8). From Fig. 8 we found the bubble luminesced immediately as an argon bubble did after the driving pressure was recovered. Compared with a fresh air bubble luminescence whose transition time scale is at the order of magnitude of second [Fig. 7(d)], a used air bubble luminesces more quickly, which is consistent with the result of Ref. [5]. While the experiments of Ref. [5] showed that the intensity (the quantity of the photons emitted from the bubble) of a rectified air luminescing bubble could recover immediately after the pressure recovered from a nonluminescing status, our experiments showed not only the intensity but also the spectra (the distribution of photons to the frequency) of a rectified air bubble recovered very quickly, which is similar to a luminescing argon bubble. However, the spectra of the rectified air bubble is not the same as that of an argon bubble. So we hypothesize that the air-SA SBSL might not rectify as much argon as air-water SBSL does.

According to argon rectification [3–5,22] and the above experiments, the gas trapped in a luminescing bubble in SA is no longer composed of fresh air. However, according to Figs. 7(b) and 8, it is obvious that spectra of a rectified air bubble and a pure argon bubble are different. Hence the gas trapped in a luminescing air bubble is not pure argon gas because there was no argon line observed in Figs. 7(d) and 8. We now want to know how much argon can be rectified and kept inside the bubble. An experiment has been conducted for this purpose. We prepared five kinds argon-air mixed gases at different volume concentrations of argon, 10, 20, 40, 60, 80 vol %, respectively. The spectrometer was set for successional shooting mode with the exposure time of 0.2 s. Before injecting the mixed gas into the host liquid (SA with 20×133.3 Pa air), the spectrometer had already been turned on to catch its onset luminescing spectrum. Figure 9 illustrates the onset spectra from air-SA SBSL of these mixed gases. The results show that the intensity of the argon atomic lines increases with its concentration. Even if the concentra-

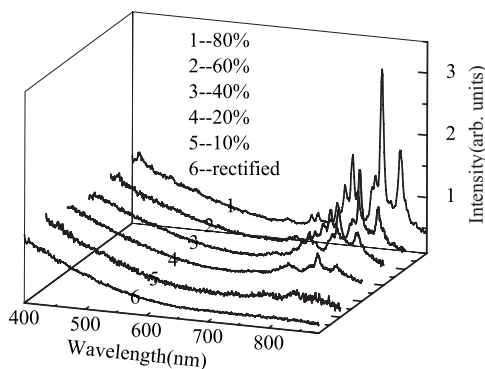


FIG. 9. The onset spectra of luminescing bubble (in 85-wt % SA) filled with argon at six different volume concentrations. Spectrum 6 comes from an argon-rectified air bubble. The driving parameters are $f=26.6$ kHz and $P_a=1.32$ bars.

tion of argon gas is at 10 vol %, the bubble still emitted distinguishable argon atomic lines (see spectrum 5 in Fig. 9). The spectrum of a rectified air bubble is also plotted in Fig. 9 as spectrum 6, where there is no atomic line. Based on these observations we conclude that the argon concentration inside the rectified air bubble is lower than 10% in our air-SA SBSL experiments, which suggests that it is weak rectification similar to the case of branch B in the air-water SBSL phase diagram in Ref. [22]. There might be a strong rectification corresponding to the branch C [22] if we boost the sonoluminescence. Unfortunately, the instability of bubble in air-SA SBSL does not allow us to try to lift the ability of rectification by increasing driving sound pressure.

In our SA SBSL the amount of the argon inside the bubble is ultimately determined by the dissolved argon concentration in the host liquid due to the mass exchange. The injected gas only gives the onset spectra, while the dissolved gas in the host liquid determines the final equilibrium spectra. In Fig. 7(a) the argon line emissions are observable in the SBSL of an injected argon bubble at the beginning. Then they fade

out gradually up to vanishing because the dissolved gas is air. A contrary case, fade-in argon lines, can be observed in Fig. 7(c). Considering the amount of argon inside the bubble, we can easily understand that the argon line emissions are always observable in the case of Ar-in-Ar [Fig. 7(b)], and not observable in the case of air-in-air [Fig. 7(d)].

V. SUMMARY AND DISCUSSIONS

In conclusion, the line emissions of OH^* [14], Na, Na-Ar*, and Ar [19] have been observed in stable and bright SBSL, which means that the line emission is no longer a distinguishing characteristic between MBSL and SBSL. The line emissions show that the bubble contains at least the vapor [6], droplets of the host liquid, and the gas dissolved in the host liquid [3]. The time-resolved spectra show that there exists matter exchange between the inside and the outside of the bubble. The transition time ranges from <0.1 s to a few tens of seconds. The SA SBSL supports the argon rectification theory [3–5,22]. Limited by the strict experimental conditions, however, we only observed the weak rectification [22] that the rectified argon concentration is lower than 10% in the SA SBSL driven by $P_a=1.22$ – 1.42 bars at $f=26.6$ kHz. The experiments show that the noble gas plays an important role in all kinds of line emissions. Even the MBSL will lose its line spectra if the host liquid is not dissolved with enough noble gas. However, the detailed function of the noble gas in line emission is worthy of further investigation.

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