Sonoluminescence of Na Atom from NaCl Solutions Doped with Ethanol

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Sonoluminescence spectra from argon-saturated NaCl solution were measured in the concentration range of 0.5−4 M at the frequency of 138 kHz. The line broadening of sodium atom emission was observed at various acoustic powers in the range from 1.8 to 16.2 W. The sodium D line showed a maximum intensity at a NaCl concentration of 2 M, which corresponded to the maximum production of OH radicals estimated by KI dosimetry. The effects of the addition of a small amount of ethanol on the line width and intensity were closely investigated at various acoustic powers. The sodium line width increases with ethanol concentration and also with power, whereas the line intensity is strongly quenched with increasing ethanol concentration. The results conclusively show that the sodium emission occurs in the gas phase within bubbles. The line broadening is due to interactions with high-pressure argon, and the maximum relative density of gas at bubble collapse was estimated to be 59.5 from the comparison with spectroscopic data. Further line broadening and quenching upon the addition of ethanol arise from collisions with gaseous products obtained from the decomposition of ethanol. The mechanism of sodium excitation is inferred to be as follows. Sodium ions enter bubbles as droplets, and salts are formed because of the high temperature within bubbles. Sodium atoms are generated by the dissociation of salts and then undergo electronic excitation by OH and H radicals.

Introduction

Sonoluminescence (SL) can be used as a spectroscopic probe of species produced at bubble collapse. Studies of multibubble sonoluminescence (MBSL) from alkali-metal salt solutions have revealed emission from the excited alkali-metal atoms.[1−10] Taylor and Jarman[3] obtained an asymmetrically broadened spectrum of the sodium D line from xenon-saturated NaCl aqueous solution. Sehgal et al.[4] performed a detailed study on sodium and potassium salts. They estimated the cavitational pressure and temperature from the broadening or shift of the emission lines, assuming that the emission arises from the highly compressed gas-phase condition within bubbles. The proposition that atomic emission originates from the gas phase is also supported by Lepoint-Mullie et al.[5] They demonstrated that the blue satellite that accompanies the broadened rubidium line is due to the B−X transition of alkali-metal/rare-gas van der Waals molecules, and claimed that the mechanism of the excitation of the alkali metal is chemiluminescence in which OH and H radicals participate. The alternative to the gas-phase origin of the emission is a liquid-phase origin. Flint and Suslick[6] reported the effects of solvent vapor pressure and an inert gas on the potassium line and observed no change in the line width or peak shift. They concluded that alkali-metal emission originates from the liquid phase and that the line broadening is caused by the rapid collisional deactivation of the excited atoms. Along the line of the liquid-phase origin, the sodium emission was interpreted by a cooperative dynamic phase transition effect.[7] In surfactant solutions such as sodium pentylsulfonate, the intensity of sodium emission was enhanced.[8] This is caused by the higher local concentration of Na^+ at the surfactant-coated bubble surface compared with that in bulk solution. On the basis of this observation, Ashokkumar et al.[9] suggested that the reduction of Na^+ occurs at the bubble/liquid interface.

It is still under debate where alkali-metal emission occurs, how alkali-metal ions are reduced, and how alkali-metal atoms are excited.[11] The purpose of the present work is to clarify these problems. We investigated the concentration and acoustic power dependences of the sonoluminescence spectrum at a frequency of 138 kHz in argon-saturated NaCl aqueous solutions. We measured the production of OH radicals by KI dosimetry to establish its correlation with the intensity of sodium emission. The effects of the addition of a small amount of ethanol on the line width and intensity of sodium emission were also studied. The results strongly indicate that the sodium emission occurs in the gas phase within bubbles.

Experimental Section

We measured the MBSL spectrum from 260 to 620 nm for NaCl aqueous solutions of concentrations in the range of 0.05−4 M at an acoustic power of 8.9 W. A narrow-band and high-resolution spectrum around the sodium emission line was measured at a fixed concentration of 2 M at acoustic powers of 1.8−16.2 W, and was also measured after adding ethanol in the concentration range from 0.5 to 2 mM at acoustic powers of 1.8−16.2 W. The experimental apparatus used for the measurement of the MBSL spectrum is described elsewhere.[12] A cylindrical cell was made of stainless steel. The size of the sample container was 46 mm in diameter and 150 mm in length. The temperature of the sample was fixed to 293 K by circulating water around the container. The top and bottom faces of the cell were equipped with a quartz glass window and a sandwich-type transducer, respectively. The solution was degassed and sparged by argon gas for 2 h. Since the sample container was closed without air, no air was introduced during the experiments. The signal from a function generator (Agilent, 33250A) was amplified using a power amplifier (NF Circuit, HSA4014) and was impedance matched to the transducer using a transformer. Emitted light was analyzed using a system of a monochromator

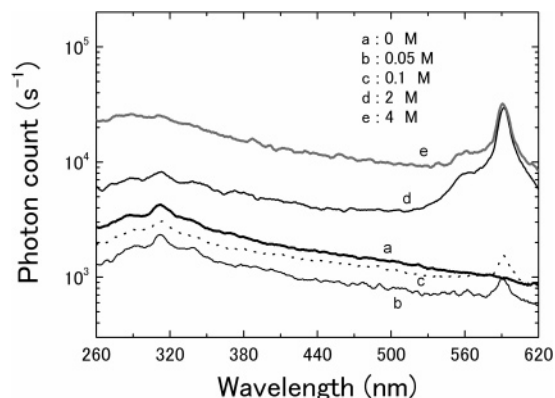* Corresponding author. E-mail: pkchoi@isc.meiji.ac.jp.

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Figure 1. Sonoluminescence spectra from NaCl solutions with concentrations of 0.05 (b), 0.1 (c), 2 (d), and 4 M (e). The spectrum denoted by "a" represents that for argon-saturated water.

(Nikon, P250) and a photomultiplier (Hamamatsu, R1527P) for broad-band measurements, and a system of a monochromator (Acton Research, SpectraPro-300i) and a cooled-CCD detector (Princeton, Pixis) for narrow-band measurements around the sodium emission line. The broad-band spectra were collected using a grating of 1200 grooves/mm blazed at 300 nm with a 1 mm entrance slit. The narrow-band spectra were collected using a grating of 1200 grooves/mm blazed at 500 nm with a 0.1 mm entrance slit. The instrumental bandwidth of the latter was estimated to be 0.315 nm from the measurement of the He−Ne laser line. The spectral response was calibrated for detection efficiency against a standard halogen lamp. The total power of the irradiated ultrasound was determined by calorimetry using a type-K thermocouple. The powers used were 1.8, 2.3, 4.8, 7.3, 9.9, and 16.2 W. The quantity of OH radicals produced was estimated by a colorimetric procedure based on the reaction with potassium iodide. The absorbance of I_3^- at 355 nm was measured for the solutions after the addition of 0.1 M potassium iodide and ultrasound irradiation for 10 min. The absorbance gives a measure of the quantity of OH radicals.

Results

Broad-band spectra of MBSL were measured for the NaCl concentrations of 0.05, 0.1, 0.5, 1, 2, 3, and 4 M. The results are shown in Figure 1 only for the cases of 0.05, 0.1, 2, and 4 M for clarity. In the case of argon-saturated pure water, which is indicated by "a: 0 M" in Figure 1, the spectrum consists of a continuum and three peaks due to the excited states of OH radicals at about 310 nm.[12−14] For the NaCl solutions, the total SL intensity, of which the continuum is the main contributor, decreases for concentrations less than 1 M and then increases at higher concentrations. The maximum amplification factor is about 7 at 4 M compared with the intensity for argon-saturated water. A sodium line with line broadening was observed in all the NaCl solutions measured. A blue satellite peak at about 560 nm accompanied the sodium line. This satellite peak has been also reported in several works.[3−5]

Figure 2 shows a summary of the concentration dependence of the intensity of sodium line relative to the continuum, as indicated by solid circles. The absorbance of I_3^- at 355 nm, which is proportional to the production of OH radicals, is also plotted as triangles in Figure 2. Both properties show maximum values at 2 M. This suggests that the OH radicals may participate in the excitation process of sodium atom emission.

High-resolution spectra of the sodium atom emission are illustrated in Figure 3 at the acoustic powers of 2.3 W (denoted by "b" in the figure), 4.8, 7.3, 9.9, and 16.2 W. The flame
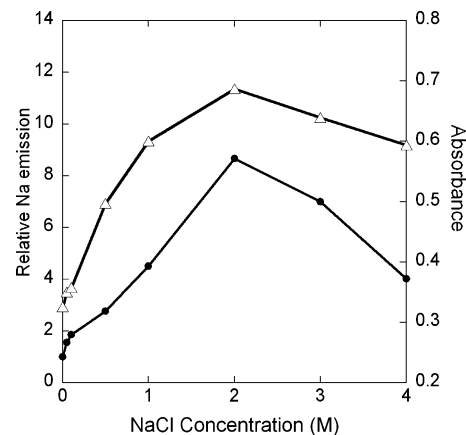

Figure 2. NaCl concentration dependence of the intensity of Na atom emission relative to the continuum (solid circles) and the absorbance of I_3^- at 355 nm (triangles), which is a measure of OH radical production.
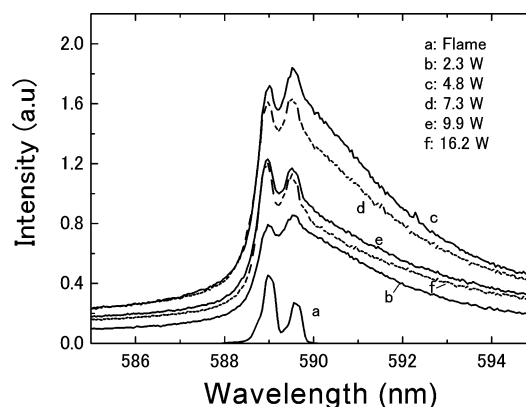

Figure 3. Spectra of Na atom emission from 2 M NaCl solutions at the acoustic powers of 2.3 (b), 4.8 (c), 7.3 (d), 9.9 (e), and 16.2 W (f). The line "a" denotes the spectrum obtained from NaCl in a flame.

spectrum of NaCl was also measured and is indicated by "a" in the figure. The peaks of the D-line doublet from the flame spectrum are at 589.0 nm ($^2P_{3/2} \rightarrow {}^2S_{1/2}$) and 589.6 nm ($^2P_{1/2} \rightarrow {}^2S_{1/2}$). The spectral peaks of SL are not shifted from those of the flame spectrum, and the lines are broadened asymmetrically to the red side (longer wavelength) compared with the lines from the flame spectrum.

The effect of ethanol molecules on sodium emission is an essential test for identifying the site of the emission, because ethanol molecules are known to evaporate in the interior of bubbles.[12,15] The spectra of sodium emission were obtained from 2 M NaCl solutions at a power of 7.3 W after addition of ethanol with the concentrations of 0.5, 1, 1.5, and 2 mM. Figure 4 shows the ethanol concentration dependence of the sodium emission spectra. In the figure, the spectra from top to bottom show ethanol concentrations of 0, 0.5, 1, 1.5, and 2 mM. The line intensity was quenched rapidly with the addition of ethanol. The line width increases with the ethanol concentration. The power dependences of the line intensity and width are discussed in the following section.

Discussion

The dependence of the MBSL spectrum on NaCl concentration is discussed in terms of two aspects: total SL intensity (the continuum) and sodium atom emission. The total SL intensity decreases slightly at concentrations below 1 M and increases at higher concentrations. Similar results were reported
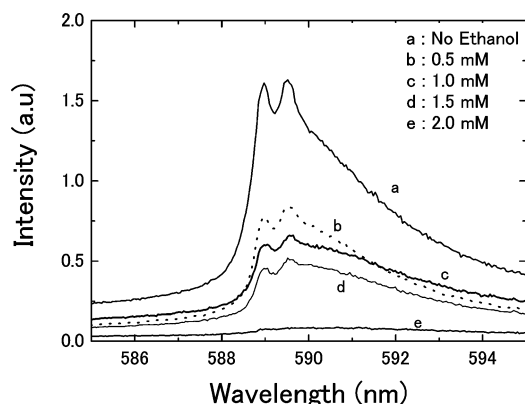
Figure 4. Spectra of Na atom emission from 2 M NaCl solutions after adding ethanol with concentrations of 0.5 (b), 1 (c), 1.5 (d), and 2 mM (e). The line "a" denotes the spectrum obtained without adding ethanol. The acoustic power is 7.3 W.

by Wall et al.,[16] who obtained SL enhancement at concentrations of up to 3 M at 515 kHz, although they did not report a decrease in intensity at lower concentrations. They attributed the SL enhancement to the reduction in gas solubility with increasing NaCl concentration. The results of single-bubble sonoluminescence by Nozaki et al.[17] also showed an increase in SL intensity at concentrations of up to 5 M. They ascribed the SL enhancement to the decrease in surface tension and vapor pressure with increasing NaCl concentration. The decrease in gas and/or vapor content results in reduced heat capacity and hence in a higher temperature within bubbles, which also contributes to the SL enhancement. Craig et al.[18] studied that bubble coalescence is suppressed at NaCl concentrations higher than 0.08 M. This suppression may cause the increase in bubble population, resulting in the SL enhancement. The origin of the small decrease in the SL intensity at concentrations below 1 M is unclear, and requires investigation on bubble dynamics.

The intensity of the sodium line, on the other hand, shows a different concentration dependence. The intensity and quantity of OH radicals are maxima at a concentration of 2 M, as shown in Figure 2. A similar strong correlation between the sodium line and OH radical production has been observed in sodium dodecyl sulfate solutions by Choi and Funayama.[19] Lepoint-Mullie et al.[5] have pointed out that the excitation mechanism of sodium atoms is through chemical reactions involving OH and H radicals:

$$OH + H + Na \rightarrow H_2O + Na* \qquad (1)$$

and

$$H + H + Na \rightarrow H_2 + Na* \qquad (2)$$
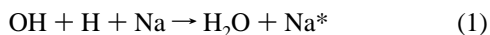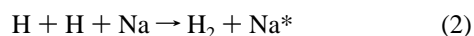
where Na* denotes the electronically excited state. These reactions have been proposed on the basis of flame spectroscopic studies.[20] The results shown in Figure 2 directly confirm occurrence of reaction 1 in the excitation mechanism. Reaction 2 may reasonably occur since OH and H radicals are produced from water vapor within the high-temperature region of the bubble core. The H radicals are, however, known to be converted to H_2 in the gas phase or to OH radicals in reaction with H_2O.[21] Thus the dominant mechanism of the excitation of sodium atoms is reaction 1. In these reactions the presence of sodium atoms within bubbles is hypothesized, and the processes by which they enter the bubbles are discussed later.

Finding the site of sodium atom emission is essential for clarifying the emission mechanism. In several works,[3-5] it has been suggested that the asymmetric broadening of the sodium line, as shown in Figure 3, is due to the interaction of sodium atom with high-pressure argon within bubbles. Extensive spectroscopic studies of the asymmetric broadening of alkali-metal emission have been carried out.[22,23] The broadening is caused by the finite difference of interaction energies in the initial and final states when the radiating atom collides with a foreign atom. There is a possibility, however, that the emission occurs in the liquid phase at the bubble/liquid interface since sodium ions are nonvolatile. Flint and Suslick[6] argued that sodium ions are reduced and excited through reactions with OH and H radicals that have diffused out of cavitating bubbles. The broadening of the line was attributed to the rapid quenching by collisions with solvent molecules. In some surfactant solutions containing sodium,[8,19] the sodium emission was observed at significantly lower concentrations than that from NaCl solutions. This is because sodium ions are electrostatically accumulated around the bubble surfaces which are adsorbed with surfactant anions. These results imply that the emission occurs close to the bubble/liquid interface.

Ethanol molecules are known to enter bubbles because of their volatile nature. In previous studies,[12,15] the addition of ethanol to argon-saturated water caused SL quenching and a decrease in the high-energy part of the SL spectrum, which suggests a temperature decrease in the bubble core. Rae et al.[24] estimated the mean bubble temperature by measuring the yields of hydrocarbon products, such as ethane, ethylene, and acetylene, obtained from the decomposition of C_1-C_5 aliphatic alcohols. They obtained a temperature of 3400 K at an ethanol concentration of 300 mM and an extrapolated value of 4600 K at zero concentration. The present results of the effect of ethanol on sodium emission, as shown in Figure 4, demonstrate that line broadening is further enhanced and line intensity is rapidly quenched with increasing ethanol concentration. The enhancement of the line broadening is explained by collisions of excited sodium atoms with the hydrocarbon products of ethanol decomposition in addition to collisions with argon gas. Norrish and Smith[25] studied the quenching of sodium emission by various gases. They showed that unsaturated hydrocarbons and H_2 strongly quench the emission, while inert gases and saturated hydrocarbons weakly quench the emission. Hooymayers and Alkemade[26] also reported the strong quenching effect of CO, O_2, H_2, and N_2 on sodium emission in a flame. They inferred that the effect is associated with the small energy difference between the alkali-metal excitation energy and the nearest molecular vibrational energy. The strong quenching upon the addition of ethanol is thus accounted for by the collisions with unsaturated hydrocarbons and H_2, which are produced during the decomposition of ethanol.[27] Another reason for the strong quenching may be the decrease in the concentration of OH radicals due to reactions with ethanol, which is known to be a typical scavenger of OH radicals. The decrease in OH radical concentration prevents the excitement of sodium atoms via reaction 1. Accordingly, the effect of ethanol on the sodium line width and intensity strongly indicates that the sodium emission occurs in the gas phase inside bubbles.

The acoustic power dependence of the sodium line width and intensity in NaCl solutions are shown in Figure 5. The closed circles denote the line width, and the open squares indicate the emission intensity. Since the spectral profile could not be fitted with a suitable function, the observed doublet of the line was numerically resolved into two lines on the assumption that the ratio of the single-line intensity is 2, which was theoretically predicted, and that the line shapes are analogous to each other.
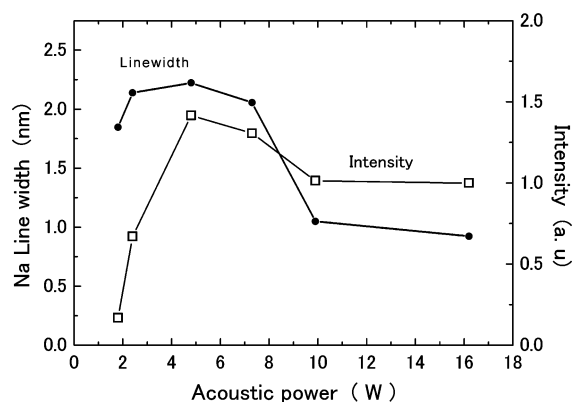
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Figure 5. Dependence of the line width and intensity of Na atom emission on acoustic power. Both the line width and intensity take maximum values at a power of 4.8 W.

In a flame study,[28] it was reported that the difference in the line widths of the D_1 and D_2 lines was about 10%. The values of the apparent line width at half-height are plotted in Figure 5. The line width and intensity take maximum values at a power of 4.8 W. The power dependence of the intensity of the continuum, not shown here, exhibited a result similar to that of the line intensity in Figure 5. The decrease in SL intensity at excessive power has often been observed,[29,30] and this is probably caused by nonspherical bubble oscillations and/or bubble coalescence, which result in the decrease in pressure and temperature within bubbles.

We can estimate the relative gas density and maximum pressure at bubble collapse from the line width by comparing it with the spectroscopic data. Jongerius et al.[28] reported the collisional broadening of the Na D_2 line for the Ar perturber to be 3.31×10^{-20} cm^{-1} (atoms cm^{-3})$^{-1}$ at 2000 K. In this study, the maximum line width is 1.89 nm (53 cm^{-1}) at 4.8 W after subtraction of the instrumental width. The comparison gives a relative density of 59.5. Here we assume a temperature at bubble collapse of 4300 K, which was estimated from C_2 molecular emission.[31] Assuming that the initial gas temperature and pressure within bubbles are 293 K and 1 atm, respectively, and a perfect gas law,[2] we obtain the pressure of 873 atm at bubble collapse. Note that the estimated pressure is proportional to the assumed temperature at bubble collapse. Sehgal et al.[4] obtained a relative density in the range from 36 to 50 for sodium emission at a frequency of 460 kHz. Lepoint-Mullie et al.[5] reported a relative density of 18 from rubidium emission at a frequency of 20 kHz. McNamara et al.[32] obtained a relative density of 19 or a pressure of 300 atm from the peak shift of Cr atom emission in argon-saturated silicone oil at a frequency of 20 kHz. Although our estimated relative density of 59.5 is larger than the reference values, the present result appears to be reasonable if we consider the difference in the experimental conditions. The ultrasonic frequency may affect the bubble pressure and temperature since the bubble dynamics depends on the frequency.[33] The frequency effect on bubble temperature and pressure at collapse has not been clarified yet.

Figure 6 represents the acoustic power dependence of the sodium line width in 2 M NaCl solutions with ethanol concentrations of 0.5 (stars), 1 (closed triangles), and 2 mM (closed circles). Also shown is the power dependence of the emission intensity for an ethanol concentration of 1 mM (squares). Since the D-line doublet was too much broadened and could not be resolved into two lines, the apparent values of total line width were plotted. The line width increases almost linearly up to an acoustic power of 16 W. This is in contrast to
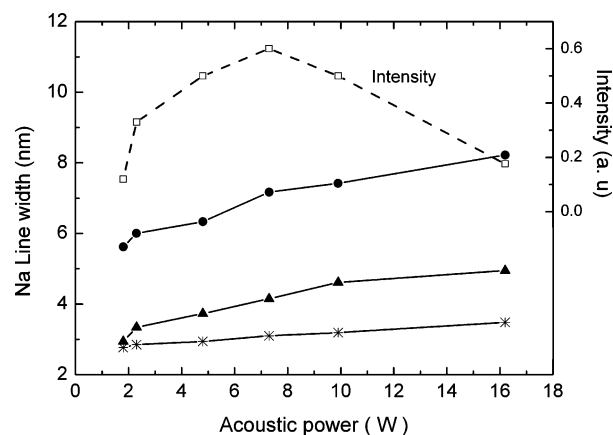

Figure 6. Dependence of the line width of Na atom emission on acoustic power for 2 M NaCl solutions with ethanol concentrations of 0.5 (stars), 1 (triangles), and 2 mM (solid circles). The intensity of Na atom emission is also represented by squares for the case of 1 mM ethanol, which shows a different dependence from the line width.

the case of no ethanol addition, where the line width takes a maximum at 4.8 W. Several types of molecule are produced from the decomposition of ethanol within bubbles. Yasui[27] simulated the bubble oscillations in aqueous methanol solution by considering various chemical reactions in bubbles, and showed that many gaseous products including H_2 and CO molecules contributed to the decrease in bubble temperature, which resulted in SL quenching. The collisional broadening of sodium lines induced by H_2 or CO molecules has been shown to be larger than that by argon.[28,34] Thus, the linear relationship between line width and power observed is thought to be explained by the collisional broadening induced by gaseous products, which increases with acoustic power. The line width shown in Figure 6 increases with ethanol concentration, and this is in accordance with the hypothesis of collisional broadening induced by gaseous products. The power dependence of the emission intensity exhibits a tendency similar to the case of no ethanol addition. This suggests that the addition of ethanol does not affect overall bubble dynamics.

It should be clarified how nonvolatile sodium ions enter the bubble interior. The salt solution evaporates into the bubble as droplets through the development of surface instabilities, which are caused by nonspherical oscillations of the bubble or bubble coalescence.[35,36] In explaining the difference in MBSL and single-bubble sonoluminescence (SBSL) spectra from NaCl solution, Matula et al.[37] indicated that the injection of liquid droplets is responsible for the sodium emission in MBSL. The SBSL spectrum exhibited no sodium line because of highly symmetric bubble collapse and no surface deformation. Using high-speed photography, Hatanaka et al.[29] observed the clustering and coalescence of bubbles in standing-wave fields, which were similar to those employed in the present experiments. Water molecules are removed from the droplets because of the high temperature during the bubble collapse phase, which leads to the formation of salt molecules followed by homolysis to generate alkali-metal atoms. The above processes are similar to those observed upon spraying aqueous NaCl solution into a flame.[38] The alkali-metal atoms are then electronically excited by OH and H radicals, as discussed previously by reactions 1 and 2.

Conclusions

Sonoluminescence from NaCl solution was observed at various NaCl concentrations and acoustic powers at a frequency

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of 138 kHz. The sodium D lines were asymmetrically broadened by collisions with high-pressure argon gas at bubble collapse. The addition of a small amount of ethanol resulted in further line broadening and in the quenching of the sodium emission, which are caused by collisions with H_2 and hydrocarbon molecules resulting from the decomposition of ethanol. The effect of ethanol conclusively shows that the sodium emission occurs within bubbles. Sodium ions enter the bubbles as solution droplets. NaCl molecules are formed and then dissociate to generate sodium atoms during the high-temperature phase of bubble oscillations. Sodium atoms are electronically excited via reactions with OH and H radicals. The maximum value of the relative gas density within bubbles was estimated to be 59.5. The study of SL at a higher frequency of 1 MHz is currently in progress to examine the effect of frequency on alkali-metal emission.

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