



Control of sonoluminescence signal in deionized water using carbon dioxide

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ABSTRACT

Megasonic cleaning is routinely employed in semiconductor industry for cleaning of wafers. However, the method also results in damage to wafer features and such damage has been proposed to arise from transient, imploding cavities formed during megasonic processing. Transient cavitation is associated with the release of light, a phenomenon called sonoluminescence (SL) and the extent of damage has been shown to correlate with the intensity of SL. Control of sonoluminescence may therefore allow control of damage during megasonic processing of wafers. In this study, the ability of carbon dioxide to quench sonoluminescence generation in deionized water exposed to megasonic field of varying power density and duty cycle has been systematically investigated. It has been found that CO₂ is not only incapable but also a potent inhibitor of sonoluminescence, providing a potential means for selective alleviation of the violent effects of transient cavitation in process fluids. A novel chemical method has been established for *in situ* release of CO₂ from NH₄HCO₃ through a pH induced shift in the carbonic acid equilibria in deionized water. Using this method, a precisely controlled, progressive decrease in SL of air saturated deionized water through addition of NH₄HCO₃ has been demonstrated. It has been determined that 130 ppm of released CO₂ is sufficient for complete inhibition of sonoluminescence generated in air saturated deionized water.

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1. Introduction

Megasonic cleaning has been traditionally used for cleaning of wafers and photomasks in semiconductor processing [1,2]. Megasonic agitation is believed to remove contaminant particles primarily through exertion of physical forces generated by acoustic streaming and acoustic cavitation [3–5]. However, cavitation also results in wafer damage [6–9]. With the advent of the sub-45 nm technology nodes, the tolerable size of contaminant particle has decreased while wafer features have become increasingly fragile, placing stringent requirements on megasonic cleaning performance. It has been suggested that damage to wafer features primarily arise from violent implosions of transient cavities while cleaning is enhanced by the streaming forces generated by stable cavitation [5]. Transient cavitation is associated with the release of light, a phenomenon known as sonoluminescence (SL) [10]. Conditions that generate high SL also lead to increased pattern damage [11,12]. Therefore, selective control of transient cavitation or the associated phenomenon of sonoluminescence may allow development of damage-free megasonic cleaning of sub-45 nm structures [7,13].

Sonoluminescence has been a subject of extensive research ever since its discovery in 1930s [14–18]. Though it is generally accepted that light is released when high temperature and pressure conditions are reached during the implosion of transient cavities, a rigorous theoretical understanding of the underlying mechanisms in SL generation and the origins of light emissions, remain elusive till date [17,19,20]. Numerous factors such as nature and concentration of dissolved gases [15,21], solutes [10,22–24], pressure [24], temperature [25], intensity and frequency of sound waves [16,24,26], second order effects arising from interaction of sound waves with solid interfaces [4,27,28], are known to influence cavitation, making it difficult to control SL generation or to deconvolute the role of individual factors in SL generation.

The successful development of a portable and UV light tight cavitation threshold (CT) cell that allows the measurement of SL under precisely controlled conditions, has recently been reported. The CT cell has been used to demonstrate the control of SL generation in air saturated deionized (DI) water through dissolved O₂ scavenging [13]. In the present work, the use of CT cell to systematically investigate the role of dissolved gases such as air, N₂, O₂, CO₂ and Ar, in the generation of SL in DI water is reported. Consistent with previous reports, these gases, together comprising 99.99% (V/V) of air, were all found to generate SL signal with the sole exception of CO₂. Based on the inability of CO₂ to support

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SL, alteration of dissolved CO₂ levels was used as a means to control SL generation in air saturated DI.

The effect of addition of CO₂ gas on the SL generation in air saturated DI water has been studied to investigate if CO₂ had an inhibitory role and if it would suppress SL generation in presence of other dissolved gases. A novel method for *in situ* release of CO₂ from NH₄HCO₃ by a pH-induced shift in the carbonic acid equilibria has been investigated. Several likely mechanisms by which dissolved CO₂ may quench SL generation have been proposed.

2. Experimental procedures

All experiments were conducted in a Class 100 micro/nano fabrication center at The University of Arizona. SL data were measured using the recently developed CT cell as previously described [13]. DI water saturated with the gas of choice was flown through the CT cell at a flow rate of 130 ml/min and exposed to megasonic field in either continuous (100% duty cycle, 50 ms pulse period) or pulse mode (10% duty cycle, 5 ms pulse period) over a power density range of 0.1–4.0 W/cm² and a frequency of 0.93 MHz. The resulting SL data were recorded in the wavelength range of 270–650 nm. All experiments were repeated 2–4 times.

Air saturated DI water samples were prepared by exposing 2 L of DI water to ambient clean room air for at least 16 h and until O₂ concentration, measured using an oxygen sensor (Rosemount Analytical model 499A DO), reached 8.3 ppm (W/W). N₂, O₂, Ar and CO₂ saturated DI water samples were prepared by bubbling the gas in 2 L of air saturated DI water for at least 2 h and until dissolved O₂ concentration reached 0.3 ppm or less except in case of saturation with O₂ where its concentration reached 39 ppm. Reported solubilities of O₂, N₂, Ar and CO₂ in DI water, at 25 °C and 1 atm partial pressure of the gas, are 41, 18, 56 and 1503 ppm, respectively, and the dissolved O₂ concentration in air saturated DI water at 25 °C is 8.6 ppm [29].

Different concentrations of CO₂(aq) were prepared using two different methods. In the first method, increasing amounts of CO₂ gas was directly bubbled into 2 L of air saturated DI water and the resulting changes in pH and dissolved O₂ concentrations were recorded to monitor increase in CO₂(aq) levels and decrease in dissolved O₂ levels. Recorded pH values were converted to CO₂(aq) concentrations using the equations of carbonic acid equilibria in water, as previously described [13]. SL data were measured as a function of increasing CO₂(aq) concentration.

In the second method, increasing amounts of NH₄HCO₃ was added to air saturated DI water containing 3 mM HCl and SL data on the resulting solutions were measured within 10 min of NH₄HCO₃ addition. pH and dissolved O₂ concentrations were recorded at each NH₄HCO₃ concentration. The pertinent ionic equilibria in this system are shown in Fig. 1. Presence of HCl shifted these linked equilibria towards the formation of dissolved CO₂. The concentration of CO₂(aq) was calculated as a function of added NH₄HCO₃ concentration using the well known values of the equilibrium constants [29] for each of the steps in the linked equilibria (Fig. 1) along with the laws of mass and charge conservation, yielding Eq. (1) through Eq. (8) with eight unknowns. These equations are:

$$\frac{[\text{H}_2\text{CO}_3]}{[\text{CO}_2]} = K_h = 1.7 \times 10^{-3} \quad (1)$$

$$\frac{[\text{HCO}_3^-][\text{H}^+]}{[\text{H}_2\text{CO}_3]} = K_{a1} = 2.5 \times 10^{-4} M \quad (2)$$

$$\frac{[\text{CO}_3^{2-}][\text{H}^+]}{[\text{HCO}_3^-]} = K_{a2} = 5.61 \times 10^{-11} M \quad (3)$$

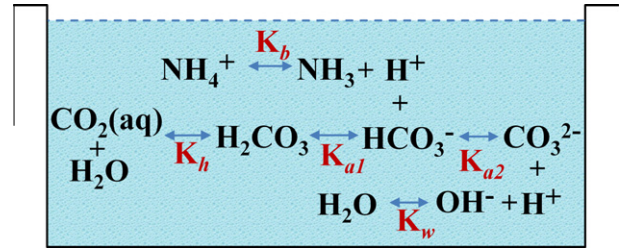


Fig. 1. Linked ionic equilibria established upon addition of NH₄HCO₃ in water. Shown are the various ionic species in equilibrium with each other and the associated equilibrium constant values (in red). K_h represents the hydration reaction constant of dissolved CO₂, K_b represents the base dissociation constant of NH₄OH, K_w represents the ionization constant of water, K_{a1} represents the first acid dissociation constant of carbonic acid and K_{a2} represents the second acid dissociation constant of carbonic acid. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

$$[\text{OH}^-][\text{H}^+] = K_w = 10^{-14} M^2 \quad (4)$$

$$[\text{NH}_4^+] + [\text{H}^+] = [\text{OH}^-] + [\text{Cl}^-] + [\text{HCO}_3^-] + 2[\text{CO}_3^{2-}] \quad (5)$$

$$[\text{NH}_4^+] + [\text{NH}_3] = C_0 \quad (6)$$

$$\frac{[\text{NH}_4^+][\text{OH}^-]}{[\text{NH}_3]} = K_b = 1.8 \times 10^{-5} M \quad (7)$$

$$[\text{CO}_2] + [\text{H}_2\text{CO}_3] + [\text{HCO}_3^-] + [\text{CO}_3^{2-}] = C_i + C_0 \quad (8)$$

Here $[\text{Cl}^-] = \text{added } [\text{HCl}] = 3 \times 10^{-3} M$, $C_0 = \text{added } [\text{NH}_4\text{HCO}_3]$ and $C_i = \text{total carbonates present before addition of } \text{NH}_4\text{HCO}_3 = 1.4 \times 10^{-5} M$. In the given equations, $[\text{H}^+]$, $[\text{OH}^-]$, $[\text{CO}_2]$, $[\text{H}_2\text{CO}_3]$, $[\text{HCO}_3^-]$, $[\text{CO}_3^{2-}]$, $[\text{NH}_4^+]$ and $[\text{NH}_3]$ are the eight unknowns, of which $[\text{CO}_2]$ and $[\text{H}^+]$ were numerically solved for, at different values of C_0 , using *Mathematica 7.0* (Wolfram Research, Inc., Version 7.0, Champaign, IL). In these calculations, the escape of CO₂(aq) from solution into the gas phase above the liquid is assumed to be negligible. The assumption is based on the low diffusivity of dissolved gases in water, including CO₂, implying that the escape of CO₂ released from NH₄HCO₃ would be negligibly small over the duration of the experiments (<15 min), if the liquid is quiescent.

DI water samples with different concentrations of dissolved O₂ were prepared by degassing air saturated DI water that was flown through the liquid port of a membrane contactor (Liqui-cel extra-flow model 2.5 × 8 inch) while vacuum pressure was applied on the gas ports using a vacuum pump (Welch Vacuum model 2545B-01). The flow rate of water to be degassed was held constant at 130 ml/min while the vacuum pulled on it was adjusted until desired degree of degassing was achieved. Extent of degassing was monitored by measuring the dissolved O₂ concentration in the degassed fluid exiting the membrane contactor using an inline oxygen sensor.

3. Results and discussion

Fig. 2 compares the intensity of SL signal in DI water saturated with air, N₂, O₂, Ar and CO₂ gas, maintained at a partial pressure of 1 atm, separately, as a function of the power density of sound waves generated in either the continuous or the pulse mode (inset). The order of SL generating efficiencies of the tested gases was found to be similar in both modes, Ar showed the highest SL intensity, followed by O₂, N₂ and Air which were all nearly the same, and CO₂ was found to be completely incapable of supporting SL generation. SL intensity of CO₂ saturated DI water was found to be within background levels throughout the applied range of

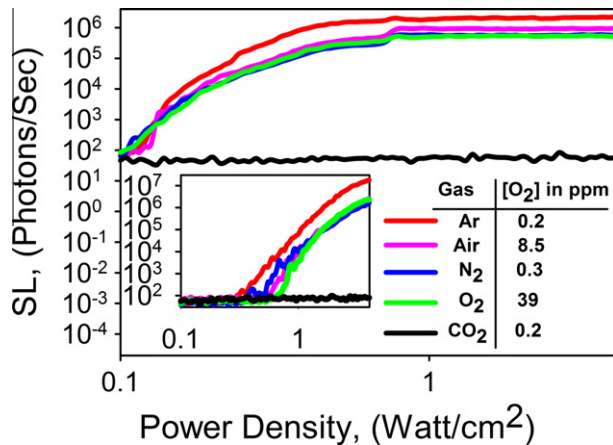


Fig. 2. SL efficiency of different dissolved gases. Pulse and continuous mode (inset) SL intensity as a function of power density for DI water saturated with different gases.

power densities and in either mode. These results are in general agreement with those reported by Young [15] and Leighton [30] except that CO₂ was reported to be weakly capable of SL generation in their studies while in this study it was found to be completely incapable of SL generation under all tested conditions. It is interesting to note that both O₂ and N₂ saturated DI water exhibited SL signal that was only ~10% lower in intensity compared to that of Ar saturated DI water. This suggests that both N₂ and O₂ are capable of multi-bubble SL generation (MBSL), as also reported in previous studies on the role of dissolved gases on MBSL generation [15].

Intensity of SL signal in DI water was found to depend not only on the power density but also the duration or % duty cycle of the sound waves driving the cavitation processes. Fig. 2 clearly shows the existence of a threshold of power density in the continuous mode (inset) that is absent in the pulse mode. Below this threshold, there is no appreciable SL generation. The cavitation threshold values for Ar, N₂, air and O₂ saturated DI water were found to be 0.3, 0.5, 0.58 and 0.7 W/cm², respectively. It should be noted that the indicated threshold, strictly speaking, corresponds to SL generation and not cavitation. However, as SL is a measure of cavitation under most conditions, the term cavitation threshold is used interchangeably with SL threshold. While SL is absent below this threshold, it is possible that cavitation, especially stable cavitation, may still exist. The dependence of the intensity of SL signal on the power density and % duty cycle of transducers may be used, in addition to its dependence on the nature of dissolved gases, for further fine-tuning of acoustic cavitation in process fluids [13].

Given the observation that CO₂ was incapable of SL generation, it was examined if CO₂ was also inhibitory for SL generation. Fig. 3A shows that the addition of increasing amounts of CO₂ resulted in the progressive decrease in SL of air saturated DI water and that the addition of as little as 60 ppm of CO₂ was sufficient to cause a near 1000-fold decrease in SL. However, the addition of CO₂ also resulted in the removal of nearly 6% of dissolved oxygen (and presumably other dissolved gases also), as indicated in the legend. This suggests that the decrease in SL may also result from the loss of other dissolved gases, rather than from CO₂ inhibition. To test this possibility, the effect of removal of an equivalent amount of dissolved gases through vacuum degassing on SL was investigated. Fig. 3B shows that the removal of up to 6% of dissolved gases by partial degassing of air saturated DI water had no significant effect on the SL generation. Further removal of dissolved O₂ below 7.9 ppm, however, resulted in a sharp decrease in SL signal (data not shown). This strongly suggested that CO₂ addition up to a level of 60 ppm indeed had a strong SL-inhibitory

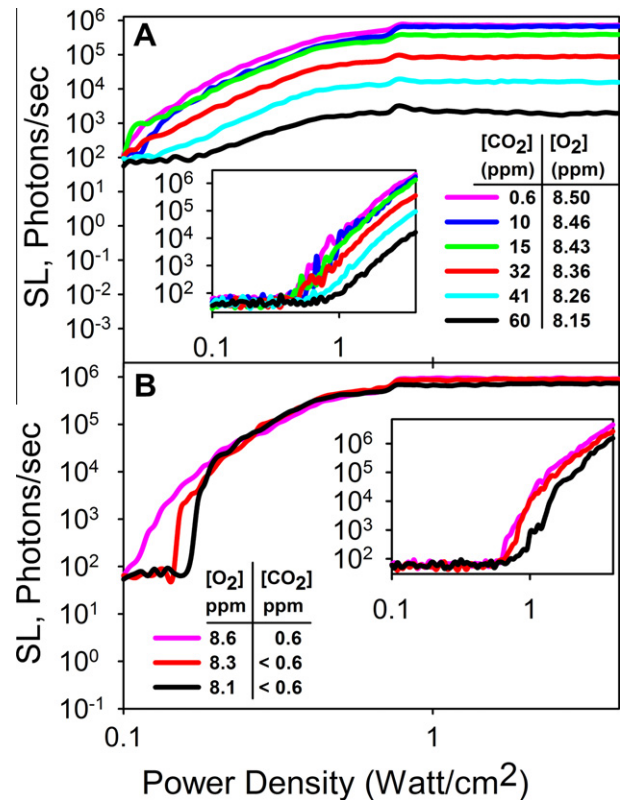


Fig. 3. Effect of CO₂ addition by bubbling on the SL generation in air saturated DI water. (A) Pulse and continuous mode (inset) SL intensity as a function of power density for DI water with different levels of dissolved carbon dioxide added by direct bubbling. (B) Pulse and continuous mode (inset) SL intensity as a function of power density for air saturated DI water from which dissolved air has been partially removed using vacuum.

effect. However, using the method of CO₂ bubbling, it was not possible to achieve complete inhibition without significant loss of other dissolved gases.

To test if CO₂ could completely inhibit SL generation in the presence of saturating amounts of dissolved air, a means of introducing CO₂ in air saturated DI water that would not alter the amount of pre-existing dissolved gases was adopted. In this method, increasing amounts of NH₄HCO₃ were added to air saturated DI water that was pre-acidified with 3 mM HCl. This resulted in the *in situ* release of CO₂ from NH₄HCO₃ that was induced by the low pH of the solution, causing the carbonic acid equilibria in the solution to shift towards formation of CO₂(aq) (Fig. 1). Fig. 4 demonstrates that while the SL intensity in air saturated DI water is progressively quenched by the released CO₂, the status of other dissolved gases is not altered at all, providing an unambiguous evidence in support of a strong SL-inhibitory role for CO₂. SL intensity of air saturated water was found to be completely inhibited by the release of 130 ppm CO₂ in solution. As shown in Fig. 4 inset, the cavitation threshold increased as SL was quenched by CO₂. Since both pH and addition of salts have previously been shown to affect SL of DI water [23], it was examined if the separate addition of NH₄HCO₃ alone or HCl alone would affect SL. It was found that neither of them affected the SL generation in air saturated DI water, suggesting that the NH₄HCO₃ induced reduction of SL in air saturated, acidified DI water is an effect of dissolved CO₂ and not HCO₃⁻ or H⁺ ions in solution.

Fig. 5B shows the theoretically calculated values of released CO₂ as a function of added NH₄HCO₃ in air saturated DI water to which 3 mM HCl was added prior to the addition of NH₄HCO₃. In these calculations, it was assumed that the released CO₂ would not un-

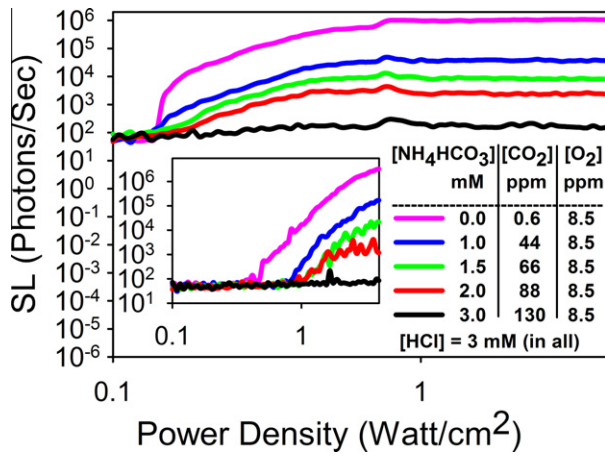


Fig. 4. Inhibitory effect of *in situ* released CO₂ on SL. Pulse and continuous mode (inset) SL intensity as a function of power density for air saturated DI water acidified with 3 mM HCl and containing indicated levels of dissolved CO₂ that was released from added NH₄HCO₃.

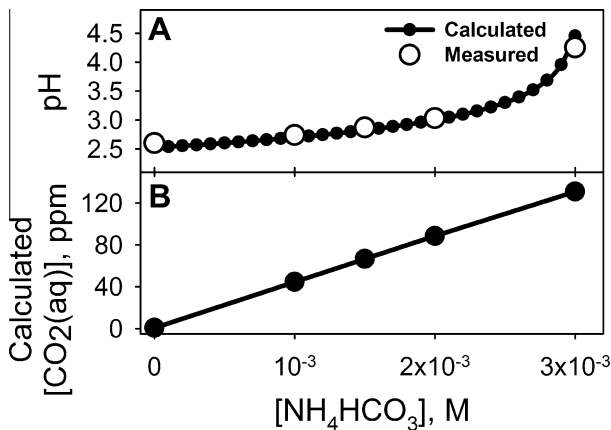


Fig. 5. Calculated [CO₂(aq)] and pH. (A) The overlay of theoretically calculated and measured pH values as a function of added [NH₄HCO₃]. (B) Theoretical estimation of the amount of dissolved CO₂ released as a function of added [NH₄HCO₃] in 3 mM HCl solution.

dergo any appreciable escape from the liquid. To test the validity of this assumption, the pH of the solution containing 3 mM HCl and different NH₄HCO₃ concentrations was estimated. If the released CO₂(aq) were to escape from the solution, the carbonic acid equilibria would be further shifted towards formation of CO₂(aq) with a consequent increase in the consumption of H⁺ ions on the other extreme of the carbonic acid equilibria (Fig. 1). This would result in an increase in the actual (measured) pH values, over and above the values calculated on the basis of no loss of CO₂(aq). Indeed the theoretical calculations were in excellent agreement with the measured pH values (Fig. 5A), thus validating the assumption of no escape of CO₂.

Though CO₂ has previously been shown to be weak in supporting SL [15,30], this work has shown CO₂ to be completely incapable of SL generation. Further, CO₂ has been found to be strongly inhibitory for SL generation and capable of quenching SL even in presence of saturating amounts of dissolved air (Fig. 4). Several different mechanisms can be proposed to explain the quenching behavior of CO₂. Being a triatomic molecule, the polytropic ratio (γ) of CO₂ is lower than that of diatomic and monoatomic gases. Eq. (9) relates the polytropic index of the gas filling a collapsing cavity to the maximum temperature, T_{\max} , reached within the cavity [18].

$$T_{\max} = T_0 \left[\frac{P_0 + P_A}{Q} (\gamma - 1) \right] \quad (9)$$

Here T_0 is the initial temperature, P_0 is the bulk solution pressure, P_A is the acoustic pressure amplitude and Q is the bubble pressure at maximum size of the bubble prior to its collapse. Treating $(P_0 + P_A)/Q$ as a lumped constant that is independent of the nature of dissolved gases and normalizing T_{\max} with this constant, Eq. (9) can be rearranged to

$$\frac{T_{\max}}{(P_0 + P_A)/Q} = T_0(\gamma - 1) \quad (10)$$

Fig. 6, which is a plot of Eq. (10), clearly shows that the normalized maximum temperature is the lowest for CO₂. Since SL strongly correlates with the bubble temperature during its collapse [15], lowering of this temperature in presence of CO₂ may therefore explain the reduction in SL intensity upon CO₂ addition. The value of $(P_0 + P_A)/Q$ is difficult to measure experimentally and depends on several factors such as the intensity and frequency of the sound waves that drive cavitation and on the extent of energy dissipation and the size distribution of pre-existing bubbles. However, it is not unusual for the value of $(P_0 + P_A)/Q$ to lie between 10 and 100 [31]. The maximum temperature reached inside a collapsing cavity in an Ar saturated water–benzene mixture, reported to be 4300 K by Didenko and Suslick [32], can be reproduced at a value of $(P_0 + P_A)/Q = 21.4$ and $\gamma = 1.67$ for Ar.

Coleman and Leighton [30] have suggested the alternate possibility that CO₂, being highly soluble in water, may cushion the collapse of bubbles through enhanced diffusion into the bubbles during its expansion phase. Chendke and Fogler [33] have suggested that CO₂ may dissipate the heat of bubble compression by undergoing thermal dissociation into CO and O₂, thereby lowering the bubble temperature and SL. CO₂ is a known free radical quencher and may inhibit SL also through quenching of luminescent free radicals that are likely to form during bubble collapse. To test this possibility, the effect of CO₂ on the SL resulting from free radicals generated in DI water using Fenton chemistry is currently being investigated.

Cavitation is widely accepted as the mechanism responsible for both cleaning and damage and hence attempts to control damage by reduction in cavitation may also reduce cleaning efficiency. However, control of transient cavitation that is associated with violent bursts of energy, without affecting stable cavitation, may primarily reduce damage. Thus, control of SL may reduce damage without affecting particle removal efficiency. Unraveling the exact

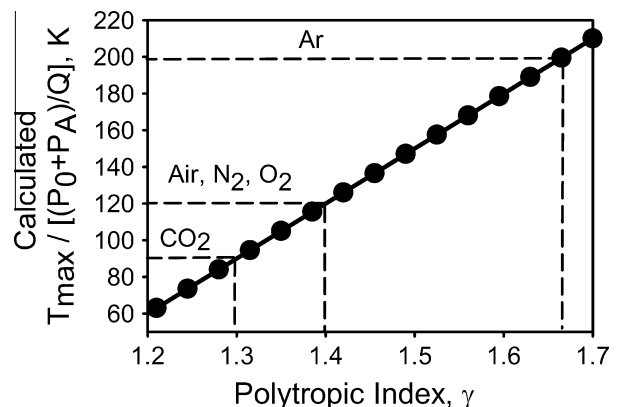


Fig. 6. Plot of the normalized maximum temperature reached in a collapsing cavity as a function of the polytropic index of the gas filling the cavity. Dotted lines show the normalized temperatures corresponding to the different gases that were studied in this work.

mechanism(s) by which CO₂ quenches SL could be key for the development of damage-free megasonic cleaning methodologies.

4. Conclusions

SL generation in air saturated DI water was shown to be strongly inhibited by addition of small amounts of CO₂. Release of CO₂ from acidified NH₄HCO₃ solution was shown to be a suitable means for well controlled and graded reduction in SL of air saturated DI water. Further studies on unraveling the mechanism by which CO₂ inhibits SL generation should be extremely valuable for development of damage-free wafer cleaning methods needed for future technology nodes in the semiconductor manufacturing industry.

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