

# Observations of bubble clusters within different cavitation environments – electrochemistry, acoustics and imaging studies

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## ABSTRACT

A set of experiments designed to characterize an ultrasonic piston like emitter and a cylindrical ultrasonic reactor are presented. These include electrochemical, acoustic and imaging of the systems deployed. An electrochemical technique, that can detect the erosion caused by single inertial cavitation events within these systems, is reported and the comparative results discussed. The technique relies on an opto-isolated measurement of erosion/corrosion of the electrode surface employed. Mapping of the systems is combined with acoustic and luminescent imaging. In addition, high-speed imaging within the different environments studied is used to support the conclusions drawn.

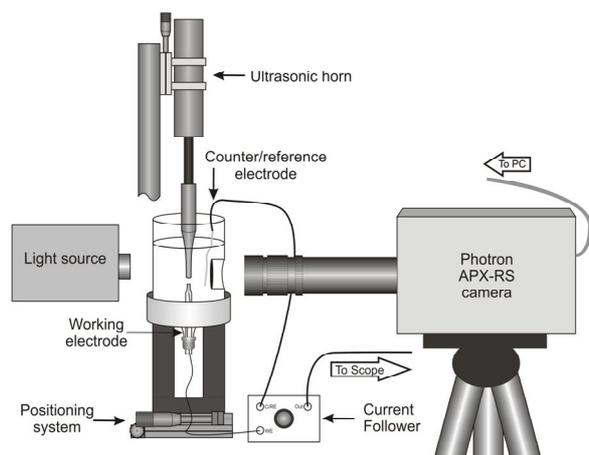
## INTRODUCTION

The environments where cavitation is produced are complex and present a significant challenge for experimental investigation [1,2]. This is as a result of their dynamic nature with the formation and destruction of bubbles and bubble clouds complicated by the acoustic interaction between the sound field employed and objects (including sensors) within the media under investigation [2,3]. The cavitation bubbles themselves contribute to the sound field by adding complexity and local effects. As a consequence many different analytical techniques, including electrochemical investigations, have been employed to probe these environments [4-8]. These electrochemical investigations date back to the 1930's [9,10]. The use of electrodes to probe the dynamics of acoustic effects has also significant lineage. For example a noteworthy study was reported by Nyborg *et al.* in the 1950's [11-13]. More recently, the measurement of pressure distributions, optical imaging both using high-speed cameras and image intensified systems as well as monitoring chemical effects have all been employed to try and characterise the cavitation environments [2,14-20]. In general the application of electrochemical techniques to this environment has been deployed to follow mass transfer effects and surface erosion produced

by the mechanisms generated by the sound field in question [5-7,21-25]. It should be noted that these electrochemical techniques can target different mechanisms within the environment. For example mass transfer of material is sensitive to forced convection within the liquid. Hence this will be sensitive to many factors including acoustic streaming, microstreaming [26-29], jetting and bubble motion. As a result of this broad sensitivity, mass transfer will be affected by inertial [30] and non inertial cavitation processes as well as the reactor geometry itself. However, surface erosion is more restrictive relying on the removal of material from a surface. This is often linked to inertial collapse close to the solid/liquid interface. Nevertheless, whatever technique is employed, advantages and disadvantages should be noted [4]. In the following discussion, a set of experiments are combined to illustrate the application of an electrochemical erosion/corrosion sensor to a number of different sound sources including a piston like emitter, a cylindrical reactor and an operating ultrasonic bath. The environments are shown to contain cluster collapse events [31-33]. The results are combined with high-speed imaging and acoustic measurements in order to support and interpret the electrochemical data.

## EXPERIMENTAL

Please note that on delivery of your manuscript you transfer your copyright on your publication to the publisher. Electrochemical measurements were performed using a two electrode arrangement. An aluminium electrode (250  $\mu\text{m}$  diameter, Advent Research materials) working electrode sealed in an epoxy support ( $\sim 2$  mm diameter) and a silver or stainless steel reference counter electrode was employed. The potential of the aluminium electrode versus the exact reference employed is shown in the appropriate figure legend. The surface of the working electrode was polished to a mirror like finish using 0.3  $\mu\text{m}$  alumina on a polishing pad. The current passed at the working electrode was monitored using a simple in-house current follower or a bespoke optocoupled current follower (details described elsewhere [34]). The data from each device was recorded using an ADC card (Measurement computing) or a Le Croy 9310 digital oscilloscope. Event counting was achieved an Amptek Pocket MCA 8000A. Ultrasound was produced using three different systems; a piston like emitter (Adaptive Biosystems, 3 mm diameter tip) operating at  $\sim 23$  kHz; a cylindrical cell [35] and a commercial ultrasonic bath (Branson B1210E-MT, 49 kHz, 80W). The piston like emitter and cylindrical cell were driven by a B&K 2713 power amplifier supplied with a signal by a function generator (TTi). In contrast the standard ultrasonic bath relied on its own power supply. An image intensified CCD video camera was used to capture images of luminescence [36]. MBSL light emission from ultrasonic bath was a composite of 101 individual images (Image Stacker software).



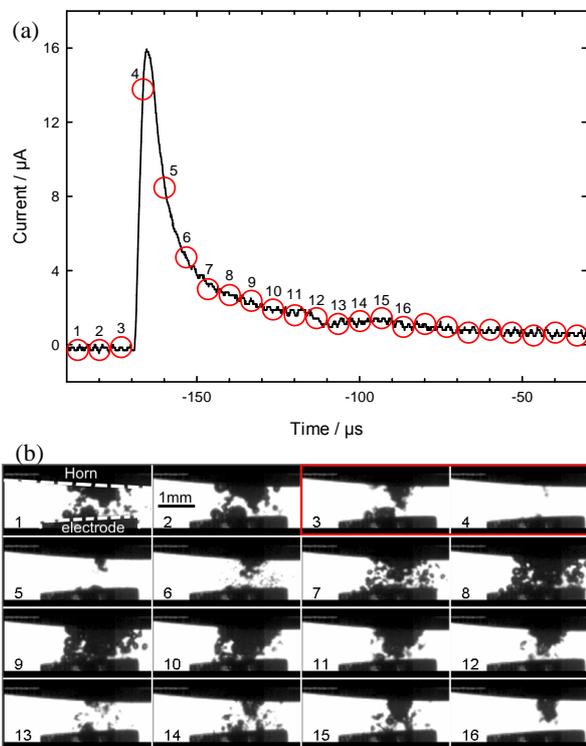
**Figure 1.** Schematic of the setup used to make high-speed images of the cavitation environment above an operating ultrasonic probe. Note here surface erosion/corrosion events are simultaneously recorded with the high-speed images obtained.

A Photron APX RS high-speed camera with a Navitar 12x lens was used to record the high-speed images. Figure 1 shows the experimental setup use to simultaneously image the cavitation environment produced by the piston like emitter while recording the simultaneous erosion signal from the electrochemical sensor. Pressure measurements were made using a Gras Type 10CT hydrophone. Sodium sulphate (Fisher, LRG) was used as received. The exact concentration is reported in the appropriate figure legend. Water was supplied by a Purite Select Fusion 160 (Ondeo) water purification system (resistivity typically  $>15$   $\text{M}\Omega$  cm and a TOC  $< 10$  ppb). Solutions were aerobic and at 20-24  $^{\circ}\text{C}$ .

## RESULTS

Figure 2 shows the electrochemical and high-speed imaging acquired for a single erosion corrosion event. Note that in the absence of erosion of the surface, no current is passed at the

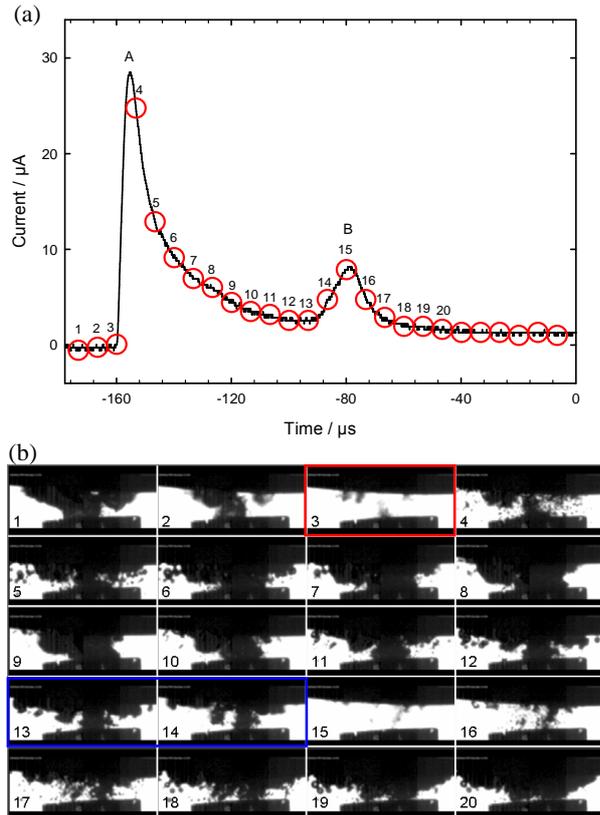
electrode. This is because of the choice of electrode material, the solution constituents and the potential control maintained on the system. However, erosion of the passive layer projecting the aluminium substrate causes an anodic current to be observed. This is due to the passivation kinetics which applies to this system. Hence the sensor response is as a result of erosion/corrosion of the solid liquid interface with the current transient recorded subsequent to the erosive mechanism.



**Figure 2** (a) Plot of a current time transient formed as a result of erosion/corrosion of a 250  $\mu\text{m}$  aluminium electrode. The numbered circles represent the duration and position of the high-speed video images (see (b)). The event was recorded in 0.1 M  $\text{Na}_2\text{SO}_4$  with the ultrasonic horn driven at 22.66 kHz and 110 V zero-to-peak. The electrode was positioned 1 mm from the tip of the horn and held at 0 V vs. an Ag counter/reference electrode. The position of the horn and electrode is highlighted in frame 1. (b) shows a set of high-speed images recorded at 150,000 frames  $\text{s}^{-1}$  with a shutter speed of 1/150000 s recorded simultaneously with the current time transient shown in (a). The red highlight shows where the cluster of bubbles has disappeared in the sequence.

Figure 2 shows that a single event can be initiated as the cluster of bubbles collapses in the space between the electrode and the horn. This is highlighted in figure 2(b) with a red oblong to indicate the temporal position of the erosion mechanism within the time window investigated. Interestingly it appears in this case that the collapse of a single cavitation bubble cannot be observed at the solid/liquid interface of the electrode under these conditions. Instead we presume that erosion is triggered as a result of cluster collapse (and associated shock generation) within the system [32,33]. The electrochemical signal from the reformation of the passive layer on the surface of the electrode can be seen to last for  $\sim 100$   $\mu\text{s}$ . During this time a new cluster of bubbles forms and starts to collapse. However, this process takes a significant time compared to the period of the sound source employed. In this experiment this period is 44  $\mu\text{s}$ . The images show that the bubble cluster survives for a least another two cycles of the sound field. In previous investigation the cluster period was found to be of the order of 3-4 periods of the sound

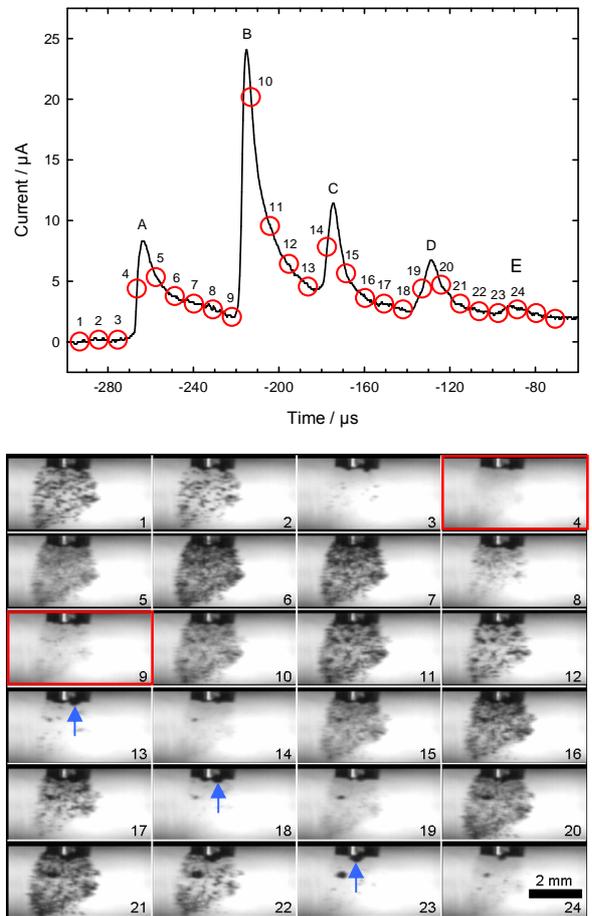
source [2]. Clearly the results shown in figure 2 indicate that the cluster collapse is of this order of magnitude. Indeed the electrochemical data shows no further erosion for at least 4 periods of the sound source. Care must be applied to the interpretation of this data. The electrochemical sensor is relatively small (250  $\mu\text{m}$  diameter) and previous investigation has shown that erosion events are not always of the same current magnitude or even detected on every cluster collapse cycle. Hence the combination of more than one technique (here high-speed imaging and electrochemical data) yields a considerable advantage in that the period and position of the cluster can be determined in addition to when erosion of the solid/liquid interface is detected. Figure 3 shows a set of data where more than one erosion event has been detected.



**Figure 3** (a) Plot of the current time trace of a ‘primary’ (A) and a ‘secondary’ (B) surface erosion/corrosion events recorded on a 250  $\mu\text{m}$  aluminium electrode simultaneously with the high-speed video images (see (b)). All experimental conditions are as shown in figure 2.

Significantly there is a considerable difference in the shape of the erosion/corrosion transient detected in this experiment. The current transient labelled (A) we define as primary and appears to be linked to cluster collapse as initiation of erosion occurs at or close to the position of frame 3 (red highlight). The current transient labelled (B) we define as a secondary event and occurs at frames 13&14 (blue highlight). Here it is possible to observe a significant bubble population within the fluid and close to the solid/liquid interface of the electrode. This is designated a ‘secondary event’ as it appears more closely linked to bubble erosion rather than cluster collapse of the collective. In addition the shape of the erosion current time transient is altered when compared to the ‘primary event’ (see figure 3 (a), ‘A’ and figure 2 (a)). The electrochemical secondary event transient is clearly more symmetric in its rise and fall. The reason for this shape change is unclear at this time although the evidence shown here suggests that secondary events are linked to a significant bubble population at the electrode surface. If inertial collapse of this bubble at the solid/liquid interface of the electrode is

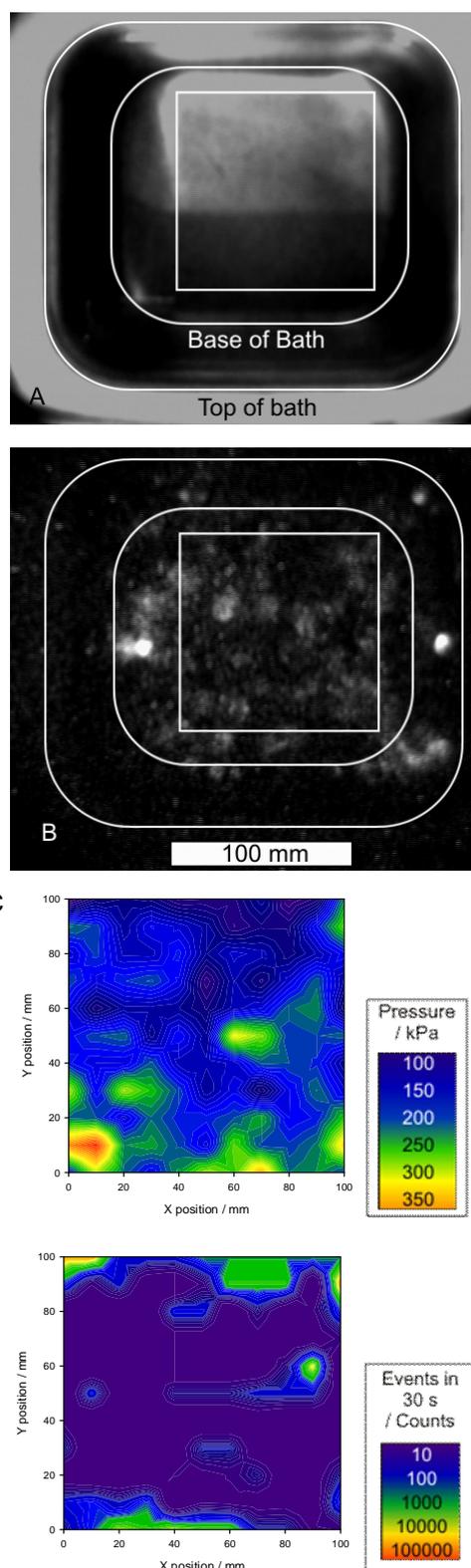
responsible for the secondary event, other effects should also be considered. For example the presence of a significant gas fraction at the solid/liquid interface has an effect on the uncompensated resistance [37] of the electrolyte. This would tend to inhibit current flow and distort the current time transient detected. It is also possible that the forces extended on the surface of the electrode are different compared to the ‘shock’ loading produced by the cluster collapse. Hence, the electrochemical properties of the media and the mechanical nature of the erosion mechanism could play roles in the transients observed. Nevertheless, the electrochemical data shows interesting additional information on the erosion phenomena that can occur within this media. Further investigations on these erosion mechanisms are currently underway. While the employment of the piston like emitter is a useful experimental tool (for example it enables a localised and controllable cavitation environment to be generated), other environments are of equal importance. For example, cylindrical reactors are useful for bulk sonochemical experiments [38-40]. Hence a number of electrochemical erosion/corrosion experiments were undertaken within this environment.



**Figure 4** (a) Plot showing the current time transients’ recorded for the electrochemical erosion/corrosion sensor (here a 250  $\mu\text{m}$  diameter aluminium disk electrode) placed within a cylindrical cell. Note two “primary” (A&B) and three “secondary” (C,D&E) surface erosion/corrosion events are highlighted. The numbered circles represent the duration and position of the high-speed video images in (b). The electrode was positioned centrally 80 mm from the base of the reactor which contain 1  $\text{dm}^3$  0.1 M  $\text{Na}_2\text{SO}_4$  and held at 0 V vs. a stainless steel counter reference electrode. The reactor was driven at 22.81 kHz with a drive voltage of 150 V zero-to-peak. (b) high-speed video images recorded at 112,500 frames  $\text{s}^{-1}$  with a shutter speed of 1/112,500 s.

Figure 4 shows the current time transients and associated bubble clouds that can be imaged within such an environment. In this system the electrode appears to be surrounded by a cluster of cavitation bubbles. Note the electrode is placed within a cylindrical cell in order to make these measurements. Hence the cluster under these conditions appears localised around the electrode rather than generated from the source (as is the case for the piston like emitter). This is of interest as the electrode itself is relatively small (~2 mm in diameter) compared to the wavelength of the sound employed (of the order of 6.5 cm but dependant on the global bubble population). Under these conditions one would not expect significant interaction between the electrode and the sound field itself. However, cluster collapse is associated with the generation of a high amplitude 'shock'. This shock will contain high frequency components which will scatter effectively off the electrode body. This scattering theory can be found elsewhere [2]. Hence the electrode is invasive for the cluster but in-invasive for the cylinders sound field itself. Turning the the erosion/corrosion events themselves, again two distinct event types can be seen in the electrochemical data. Figure 4 (A, B) represent 'primary' erosion which is associated with cluster collapse in frames 4 and 9. The time period between frame 4 and 9 corresponds to 44  $\mu$ s which is close to the period of the sound field in tis case (43.8  $\mu$ s). Again the primary event is characterised by a sharp current rise followed by a current decay over ~100  $\mu$ s. However, in this ultrasonic environment, further erosion occurs before the surface is fully passive (see frame 9, erosion transient B). While event A and B are assigned as 'primary' (corresponding to erosion of the surface associated with cluster collapse and shock loading of the solid/liquid interface), the three subsequent current time transients (C, D & E) appear different in shape and can be assigned as 'secondary'. In order to understand the cause of this difference it is instructive to look at the high-speed images shown in figure 4 (b). Again the cluster collapse is apparent (see frames 13, 18 and 23) at a period approximating to the sound field within the cylindrical reactor. However, one key difference is observable. At the electrode there appears to be a gas bubble (highlighted with a blue arrow) in each case. This bubble situated close to the solid/liquid interface appears to be a contributory factor to the change in shape observed in the current time transients. In addition, the bubble position also appears to be a contributory influence with the smallest event (E) detected when the bubble was central in the frame (compare frame 13 with frame 23, figure 4). Again it is difficult to fully assign the exact mechanism responsible for the change in event shape (from either the forces applied to the surface and/or the uncompensated resistance effect). Nevertheless, the observations in the piston like emitter and the cylindrical cell do suggest that similar erosion mechanism are present in both environments. The erosion sensor is clearly a useful addition to the range of experimental techniques which can be applied to a cavitation environment. Probably the most common ultrasonic cavitation environment is the cleaning bath. In order to investigate this environment a series of experiments were carried out on an operating ultrasonic bath. In particular the luminescence from multibubble sonoluminescence (MBSL), pressure field and erosion signal was investigated. Figure 5 shows a collection of the results obtained. Figure 5 (A) shows the bath under normal conditions (illuminated) while (B) shows the corresponding MBSL imaging. Note that this data is the sum of 101 individual frames as the individual images and luminescent output is relatively weak under these conditions. Nevertheless image (B) shows that there are areas of high cavitation activity. In addition, the field produced from the bath seems 'diffuse' in nature without a strong modal structure. Turning to the pressure mapping shown in figure 5 (C). Clearly the pressures generated by this device are significant as pressure amplitudes in excess

of 100 kPa were observed. Hence one would expect to proce inertial cavitation events (assuming suitable nuclei are present) in many positions within the bath.



**Figure 5** Ambient light (A) and intensified (B) images of the bath. The scale bar is taken for the base of the bath. (C) shows a pressure contour map from a hydrophone scanned over an area 100 by 100 mm at a resolution of 10 mm (see (B) white box). (D) shows the number of erosion corrosion events recorded on a 250  $\mu$ m Al electrode held at 0 V vs. a SS counter/reference electrode using an MCA collecting over 30 seconds (threshold of 0.85  $\mu$ A) recorded in the same positions as (C). The bath contained 1.5 dm<sup>3</sup> of 0.1 M Na<sub>2</sub>SO<sub>4</sub>.

However, the key point here is that in order for an inertial event to be generated two basic conditions have to be met. First, the pressure amplitude must be sufficient. Second, a suitable bubble nucleus is necessary. These conditions were reported by Apfel using a simple model that enables the inertial pressure threshold to be estimated for a range of bubble sizes [30,41]. Briefly, under the conditions employed, it would be expected that the minimum pressure amplitude required would be of the order of 120 kPa assuming the presence of an optimum bubble nuclei. While the pressures present within the ultrasonic bath appear impressive, the lack of MBSL output suggests that the number of suitable bubble nuclei in this ultrasonic environment are limiting. This is supported by mapping the erosion of the electrode over the same plane in the ultrasonic bath (note in both cases the hydrophone and electrode were scanned in the same plane at half depth in the bath itself). Figure 5 (D) shows the erosion mapping. In order to deploy the sensor in this particular environment an opto coupled current follower had to be deployed. This is described in detail elsewhere [34]. Although some hotspots are seen in the erosion mapping, in much of the scan little activity was observed. Clearly the local conditions are not suitable in this environment to observe widespread erosion. This supports the MBSL data for this system which also indicates sporadic inertial activity.

## CONCLUSIONS

The combination of electrochemical data with acoustic, luminescent imaging and high-speed camera observations provides valuable insight into the complex cavitation environments which are commonly employed. Erosion is particularly interesting as it appears associated with more than one mechanism. Evidence for shock erosion (associated with cluster collapse) is seen and is associated with 'primary' erosion events, while the presence of bubbles at the solid/liquid interface appears to produce different current time histories denoted as 'secondary' events. In addition the fields generated by an ultrasonic bath are impressive from an acoustic pressure amplitude perspective but can for the bath shown be relatively inactive for erosion/MBSL generation.

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