

Opto-Isolation of Electrochemical Systems in Cavitation Environments

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An electrochemical technique that can detect inertial cavitation within an ultrasonic reactor is reported. The technique relies on the erosion and repassivation of an oxide covered electrode (specifically aluminum). The sensitivity of the technique (<46 fg per erosion event) is significantly greater than normal weight loss measurements. A novel opto-isolation system is discussed which enables the electrochemical measurements to be undertaken within an earthed metallic container. Events detected in this manner are reported and compared to the noise in the absence of appropriate isolation. This system is combined with a multichannel analyzer to map the erosion/corrosion activity within an operating ultrasonic bath.

The generation, detection, and quantification of bubble related processes are of fundamental importance in many industrial, laboratory, and natural environments.^{1–7} Among these processes, cavitation is of particular interest because of the number of possible exploitative mechanisms that can be harnessed through this phenomenon. However, the quantification of cavitation is by no means trivial. While cavitation has a number of different effects including the destruction of organic materials,⁸ the emission of light,⁹ the generation of acoustic

emissions,¹⁰ the processing of polymer matrixes,^{11–13} and the cleaning/erosion of a surface, it is a particularly difficult phenomenon to quantify. This is due to the measurement strategies relying on secondary effects related to this phenomenon. Interestingly, the primary driving force for cavitation is the local pressure¹⁴ within the medium in question, while the measurement techniques usually rely on effects of cavitation on the local environment. For example, measurement strategies may include the erosion of material from a suitable substrate,¹⁵ the emission of light from the liquid,¹⁶ the acoustic emission or the generation of chemical products.^{17,18} Clearly all these approaches will have advantages and disadvantages which have been documented.⁷ However, none as yet are universally acceptable or directly measure the global response of the phenomena, rather a specific product of its action. Nevertheless, different measurement strategies may be appropriate for the particular application of the technology in question. It is the focus of this manuscript to investigate how one particular quantification technique, related to the erosion of material from a surface, may be employed in a variety of different environments. In this context, erosion of material from a test specimen can be used to estimate the erosive cavitation activity of a particular piece of apparatus. This erosion can be quantified using a simple mass loss principle.¹⁹ The detection limit of this technique will be clearly defined as the resolvable mass loss of the material compared to parasitic processes (such as corrosion) of the substrate within the media in question. Mass losses of the order of 1 mg are quoted in the literature.¹⁹ Alternatively film erosion may also be employed.¹⁴ While these

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techniques may be of use, it is difficult to see how they can be made transferable and absolute.

The use of electrochemical methods for the investigation of the effects of cavitation has been reported over the last 10–15 years^{20–24} although electrochemical investigations within acoustic fields can be found since the 1930s.^{25–28} It is important to note that the employment of electrochemical technology alone is limited in its power to solve the often complex issues related to the cavitation process. Hence, it is desirable to employ a number of other techniques, in parallel with the electrochemical investigations, to enable a better understanding of the system. In brief, it has been shown that the environment below an operating ultrasonic horn is extremely complex. Nevertheless, a useful electrochemical technique is the employment of surface erosion/corrosion measurements to determine the location of the boundary between regions where inertial cavitation exists and regions where it does not.^{29–32} In this technique an electrode is held under potential control in a passive state to form an insulating oxide or insoluble salt layer.¹⁵ In the absence of any erosion mechanisms essentially no current is observed. However, in the presence of erosion, depassivation occurs, and this is immediately followed by a repassivation step under electrochemical control. Figure 1 shows a schematic of the erosion mechanism superimposed on the current time transient detected by the electrochemical apparatus. Included in this Figure is the integration of the current transient over the first 80 μs . The transient over this time period yields 4.56×10^{-14} grams of Al_2O_3 . This figure obviously reduces for smaller transients (see later results). The sensitivity of this technique is many orders of magnitude greater (in terms of monitoring mass loss) than normal “mass loss detectors” which operate on the milligram scale.^{19,33,34} Another significant advantage of this technique is that the small electrode dimensions employed (in the case of Al electrodes, 250 μm diameter) ensures that individual cavitation events are likely to be detected. Furthermore, the fast repassivation kinetics of the films employed (typically $\sim < 200 \mu\text{s}$) enables the electrode to heal rapidly and be ‘ready’ for the next event (an enhancement in terms of temporal resolution of many orders, perhaps up to 6–8 [note, this will be dependent on the materials employed] orders of magnitude compared to mass loss experiments). We report here an extension of this technique.

However, in many electrochemical investigations, ideal conditions (from an electrochemical standpoint) have been employed. For example the tip of the piston-like emitter is isolated from the

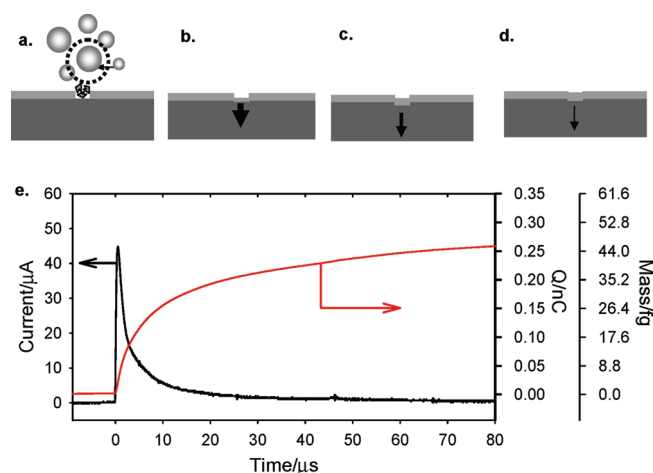


Figure 1. Images a–d depicts a schematic of cavitation induced erosion/corrosion event at a passivated electrode surface. Image a shows a bubble event causing damage to the surface of the passive film while images b–d show the electrochemical “healing” of the surface back to the passive state. Note the arrows indicate the magnitude and direction of electron flow as the passive film reforms under electrochemical control while (dark rectangle) indicates the base electrode material and (light rectangle) the passive surface. Image e is a corresponding current time trace (black line) recorded at a 250 μm diameter Al electrode. In this case the cavitation event was produced by an ultrasonic horn (23.17 kHz, 56 W cm^{-2}).³² The separation between the horn and the electrode was 0.5 mm. The electrode was held at 0 V vs Ag. The cell contained 0.25 mol dm^{-3} Na_2SO_4 . Included in the figure is the integration of the transient (red line) over this time period and the equivalent mass of Al_2O_3 produced (fg, see right axis).

system (or its potential is controlled) so that it does not interfere with the electrochemical system employed. While this is a successful strategy, it limits the apparatus that can be employed within the system. Hence, a more robust approach must be adopted before the technology can be exploited to its full potential. Consider, for example, the ubiquitous ultrasonic bath. This piece of apparatus is employed in many laboratories to clean substrates prior to further processing. These baths almost always employ a large stainless steel vessel to act as the liquid container which (as will be shown here) interfere with electrochemical measurements if directly employed in the steel vessel. Hence, to progress with an electrochemical detection strategy, it is necessary to develop a new approach within the apparatus/environment in question. To achieve these targets, we describe here an optically isolated current follower system which has suitable response times for the environments in question and will be shown to be able to record current time transients and map inertial cavitation activity within an operating ultrasonic bath.

EXPERIMENTAL SECTION

Aluminum electrodes were fabricated by sealing wire (Advent Research materials 250 μm diameter purity: Al 99.98%) inside a Pasteur pipet using slow set epoxy resin (Struers Epofix). The 250 μm diameter electrode was chosen as it represents a compromise between ease of fabrication, the shape of the resultant current time transient produced by erosion, and its relative size with respect to the erosive event produced by ultrasound in the

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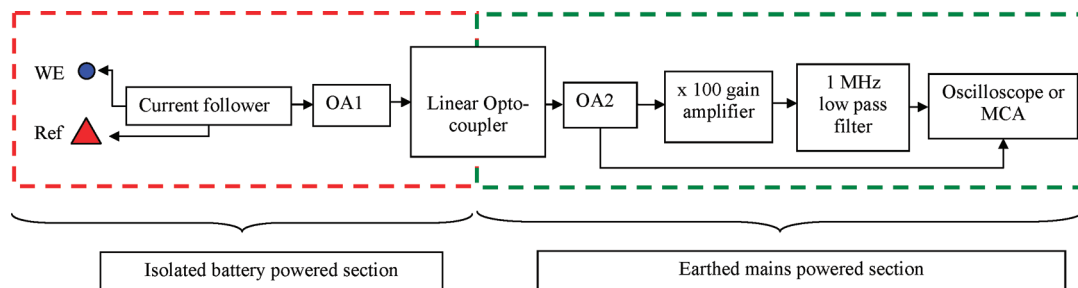


Figure 2. Schematic showing the opto-isolation apparatus, the electrochemical cell, and the recording equipment. Note the red section corresponds to the isolated current follower while the green section, the mains powered section. Here OA1 and OA2 correspond to appropriate op-amps.

environments in question.^{32,35,36} Connections to all electrodes used shielded cable to reduce electrical noise from the ultrasonic equipment. The electrodes were polished to produce a smooth reproducible surface with successive grades of silicon carbide paper (400, 600, and 1200 Deer) and an alumina slurry (1 μm Buehler) supported on Microcloth (Buehler).

To simplify the electrochemical apparatus employed, the electrodes were held at 0 V versus a stainless steel needle (19 gauge) or silver wire (0.5 mm diameter Advent research materials) counter/reference electrode. The potential of the stainless steel (SS) and Ag wire versus a standard calomel electrode (SCE) in 0.25 mol dm⁻³ Na₂SO₄ was found to be -160 mV and +200 mV, respectively. These pseudo reference electrodes were used as received. For most measurements a simple in-house current follower was used to amplify the signal. Current time transients were then recorded using a LeCroy 9310AM oscilloscope, and the data transferred to a PC.

Figure 2 shows a schematic of the instrumentation developed to measure the erosion/corrosion events in a “non-isolated” electrochemical environment. In general this consists of a current follower isolated using an opto-isolation strategy. This isolated instrumentation consisted of two sections; a simple current follower with gain adjustable from 10³ to 10⁸ V A⁻¹ and the isolating circuitry (note a post isolation amplifier and filtration circuitry is also added to improve the signal for some systems studied). Power was supplied to the isolated portion of the equipment by a 6 V lead acid battery (Maplin Aino Micro AM 6–4.5, 4.5 A h) and to the earthed portion of the current follower by a 9 V mains power adapter. Separate DC/DC converters (Tracor Power TEL 2-0521) for each portion of the equipment created a stable ± 5 V supply to the appropriate portion of the circuit. The isolation circuit was designed such that the only connection between the electrochemical circuit and the earthed environment was through an optically isolating linear opto-coupler (IL300). The linear opto-coupler used consisted of an efficient AlGaAs LED emitter coupled to two independent PIN photodiodes. In addition to one of the photodiodes transferring the signal from the electrochemical circuit, the second photodiode ensured the linearity of the output using a simple feedback circuit. This arrangement was adapted from the comprehensive application notes provided by the manufacturer.³⁷ It was found that to produce an accurate response from the circuitry it was important that the op-amp

used to drive the opto-coupling chip was capable of driving relatively large currents required (a minimum of 5 mA). If this was not achieved the circuitry was seen to “ring” and behave in a non ideal manner. To achieve this current prerequisite a TS921IN op-amp was found to be suitable. No similar requirement for the output op amp was required, and a standard op amp was used. After the opto-isolation circuit, an optional 100 \times amplifier was included to amplify the signal in addition to a switchable 1 MHz low pass filter to remove high frequency noise.

Three different sources of ultrasound were employed. First, an Adaptive Biosystems ultrasonic horn operating at 23.17 kHz was employed (~ 56 W cm⁻²).³² This produced a localized and repeatable region containing inertial cavitation (in a region close to the face of the emitting surface). The electrode could then be positioned accurately (apparatus described elsewhere³²) with respect to this sound source. The effects of cavitation and the subsequent erosion/corrosion processes could be studied. Second, an Ultrawave U50 ultrasonic bath was used. The ultrawave bath is a standard, earthed commercial bath and as such is representative of similar such systems. The bath had a volume of 600 cm³ and operates at 60 kHz. Third, a Semat ultrasonic bath with a fundamental frequency of 46 kHz and a maximum volume of 3 dm³ was used to investigate the mapping ability of the systems developed and reported here.

To count the number of events taking place on the surface of the electrode two methods were employed. In line with previous studies a fast PC based ADC card data acquisition system (Measurement Computing PCI-DAS 4020/12) was used, and the time/current data analyzed using software written in house (VB 6). In addition an Amptek Pocket MCA 8000A Multi Channel Analyzer (MCA) was employed to analyze the signal from the electrodes. The MCA records the number of peaks as a function of the magnitude of the peak. Pressure measurements were conducted using a Gras type 10 CT hydrophone attached to a Brüel & Kjaer 2635 charge amplifier and recorded using a fast PC based ADC card (Measurement Computing PCI-DAS 4020/12). To map the inertial cavitation activity with the ultrasonic bath a GPIB controlled XYZ positioning stage (ICS Electronics USB-GPIB interface connected to a Parker Digiplan 1185–100 GPIB interface card controlling three Parker CD20 Stepping Motor Drives driving 3 Unislide steppers with a 450 mm travel in each axis) was interfaced with either the MCA (surface event activity) or ADC card (pressure measurements) using software written in house (VB).

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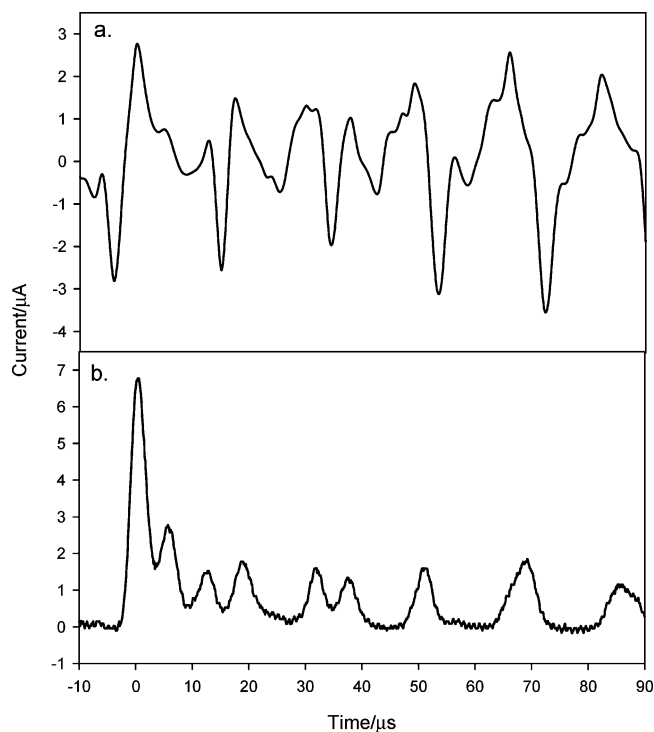


Figure 3. (a) Plot showing the electrochemical signal recorded for a 250 μm diameter Al electrode held at 0 V vs SS in an operating ultrasonic bath (Ultrawave U50). (b) Plot showing the electrochemical signal corresponding to erosion/corrosion transients recorded with the opto-isolation system described in the text. The ultrasonic bath contained 500 cm^3 0.25 mol dm^{-3} Na_2SO_4 under aerobic conditions.

All experiments were carried out in Na_2SO_4 solution (see figure legends for exact details) (Fisher Scientific, Laboratory reagent grade), the water used was purified to $>15 \text{ M}\Omega \text{ cm}$ using an USF Elga Purelab Option E10 purification system. All chemicals were used as received. All experiments were performed at 20–25 $^\circ\text{C}$ under aerobic solutions.

RESULTS

The electrochemical response of the erosion/corrosion sensor within an operating ultrasonic bath in the absence of an isolation technique is shown in Figure 3a. Note that in this experiment the electrolytic solution is in direct contact with the metallic steel container of the ultrasonic bath employed. Here it can be seen that the response of the system is dominated by both positive and negative current transients. In addition, these transients occur at a period of $\sim 16 \mu\text{s}$ which corresponds to the period of the ultrasonic source (here a 60 kHz ultrasonic bath) employed. Clearly as the system erosion/corrosion sensor is known to produce positive transients as a result of mechanical breakdown of the passive oxide film,³⁸ it is expected that only positive transients should be observed. These may occur within this current time trace but are masked by the pickup from the ultrasonic bath itself. Hence, to use the erosion/corrosion sensor within this or other similar environments, it is necessary to avoid this pickup from the ultrasonic bath. To achieve this goal, an opto-isolation system was employed (see Experimental Section for

details). Figure 3b shows a current time trace recorded for the same electrode in the same ultrasonic bath but with the application of the opto-isolation equipment. In this case the current time transient is dominated by anodic transients as expected. Figure 3b shows a series of current time traces recorded for this aluminum electrode. Interestingly some of the transients recorded can occur at time separations $<16 \mu\text{s}$. This implies that the dynamics of the erosion events may lead to erosion mechanisms at periods other than that dictated by the period of the ultrasonic source employed. This has also been observed with other ultrasonic sources other than the bath employed here.^{29,39–41} In these studies (with, for example, an ultrasonic horn), the erosion process was found to follow the periodicity of cluster events produced by the sound source.²⁹ The results shown here also indicate that the erosion mechanism within this environment (specifically an ultrasonic bath) are more complex than bubble events produced on a cycle to cycle basis. However, Figure 3b clearly shows that the apparatus developed and described here is able to measure these events within the challenging environment of an ultrasonic reactor. Clearly this is an important step for the development of this sensor for the characterization of cavitation erosion/corrosion as a means of quantifying this phenomenon. Some limitations will undoubtedly apply. For example, the quoted bandwidth of these linear opto-isolators (200 kHz) implies that the events shown in Figure 3b will be limited by this response. Indeed the current time transients recorded with a “fast” current follower in an isolated electrochemical cell (see Figure 1) are clearly sharper than the events in Figure 3b. However, the response is suitable for event counting within this environment. It is also possible that, provided that anodic transients are required, this technology may have other uses apart from cavitation erosion/corrosion. It should also be noted that because of the relatively small masses of materials involved per event, the electrochemical erosion/corrosion technique is many (up to 10^{11} as a single event compared to loss from an entire sample) times more sensitive than the simple mass loss experiments, can be used to spatially resolve cavitation action,^{29–32} can be transportable, and has a far better temporal resolution (up to 10^8 times compared to normal mass loss experiments). In addition it should be possible to deploy this apparatus in other environments where electrochemical interference from other electrodes is problematic. Note it is also possible to construct a bipolar system using this technology. However, the response time of this system is limited to $\sim 2 \text{ kHz}$.^{37,38}

To illustrate the versatility of this technique and measure the activity of an operating ultrasonic source, a series of experiments were performed to investigate whether this approach could be employed with a standard multichannel analyzer (MCA). Here a 250 μm diameter aluminum electrode was placed under an operating ultrasonic horn at a variety of source-to-electrode distances ensuring that the electrode was within the inertial zone generated by this device. The electrochemical signal recorded as a result of the erosion/corrosion events was then recorded using an ADC card and a multichan-

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Table 1. Collection of Parameters Used to Validate the MCA Analysis of the Erosion/Corrosion Events Detected by an Al Electrode^a

experiment	horn to electrode distance		trigger threshold			MCA count	ADC card count
	/mm	/bin	/mV	/μA	/counts	/counts	
1	1	75	91.6	9.2	57	49	
2	1	75	91.6	9.2	65	44	
3	1	75	91.6	9.2	4	1	
4	1	75	91.6	9.2	7	6	
5	0.5	75	91.6	9.2	17670	24112	
6	0.5	75	91.6	9.2	91496	85071	
7	0.5	200	244.1	24.4	44357	45447	

^a See Figure 4 legend for further details.

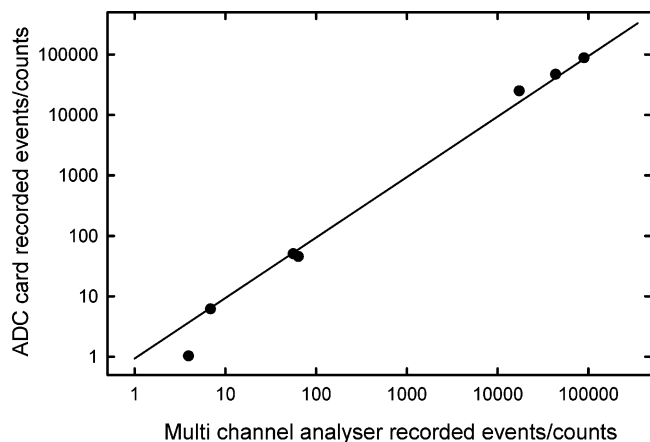


Figure 4. Plot showing a comparison of the number of erosion/corrosion events recorded by an electrode as determined by analysis of the current time data captured using an ADC card and analysis of the same current/time data with a multichannel analyzer. The data was obtained concurrently over a period of 30 s at various distances away from the tip of an ultrasonic horn to give an indication of the validity of the MCA technique at a number of activity levels. The data was recorded from a 250 μm diameter Al electrode held at 0 V vs a SS counter/reference electrode in 0.1 M Na₂SO₄.

nel analyzer for 30 s. The data recorded by the ADC card was then analyzed using bespoke software (VB). The number of erosion/corrosion events was then compared to the data obtained from the MCA. Table 1 shows a comparison of the two approaches to data analysis. Clearly, the number of events detected using both methods are in close agreement. Figure 4 shows the linearity of the two approaches over the data range investigated. Indeed this approach can be seen to be approximately linear over 4 orders of magnitude of the number of events detected. This is significant as the MCA analysis approach is useful in acquiring and analyzing the number and distribution of erosion/corrosion events produced by cavitation. This is due to the vastly smaller amount of resultant data (typically ~22 kB for the MCA vs 250 MB for the ADC) produced by each experiment and improved data processing time of this approach. This experimental approach was used to investigate the reproducibility of the technique for monitoring cavitation erosion/corrosion events. Here a 250 μm diameter Al electrode was held at +0.2 V versus SS and the number of event recorded using the MCA for 10 s (threshold level 8.5 μA). Under these conditions the number of events

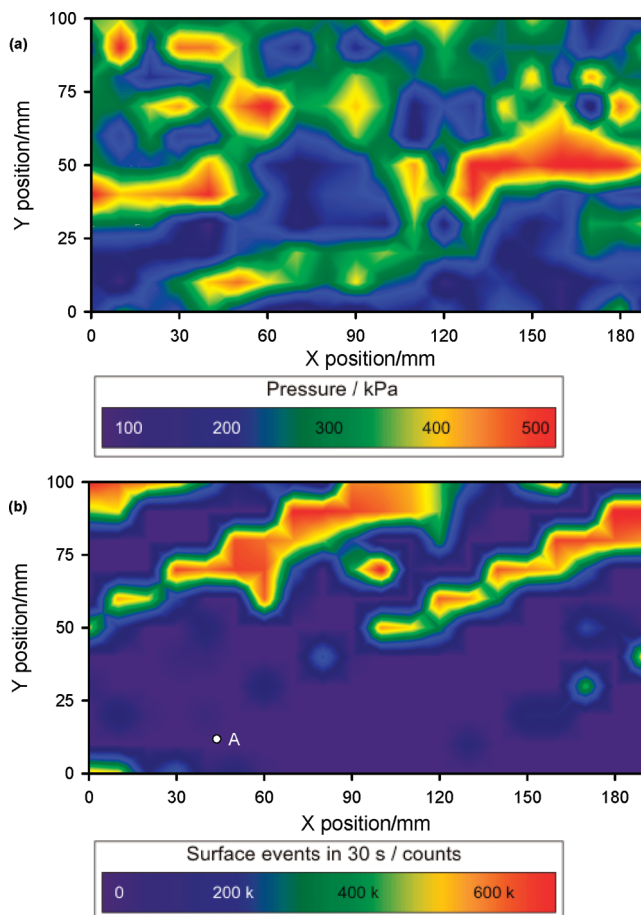


Figure 5. (a) Contour map showing the zero-to-peak amplitude pressure variation within a Semat ultrasonic bath. The hydrophone was scanned over an area 190 by 100 mm at a resolution of 10 mm in 2 dm³ of 0.1 M Na₂SO₄ solution. (b) Contour map showing the distribution of surface erosion/corrosion events over the same area with a resolution of 10 mm. Events recorded on a 250 μm Al electrode held at 0 V vs a SS counter/reference electrode using an MCA collecting over 30 s with a threshold of 0.85 μA. Note point “A” refers to a position where little or no erosion/corrosion events are detected while acoustic pressure amplitudes above the inertial threshold are expected. In each case the bath contained 2 dm³ of 0.1 M Na₂SO₄.

detected was 4187 ± 2363 per 10 s interval (95% confidence over 10 repeat measurements) and indicate in part some degree of variability that is understandably associated with cavitation. In addition the number of events was also investigated as a function of potential. Here the potential of the electrode was varied from -0.4 V to +1.0 V versus SS. However, the number of events detected in this time period was in the range expected for this ultrasonic source and under these conditions. These observations indicate that it is appropriate to select the SS electrode as a suitable reference potential. In addition the results gathered thus far indicate that the electrochemical erosion/corrosion technique, the MCA and the opto-isolated apparatus can be deployed in a scanning operation where the electrode is used to monitor inertial cavitation activity in an operating ultrasonic bath. Figure 5 shows the results of such an investigation. Here, Figure 5a shows the results of a scan of the acoustic pressure amplitude over an area of 100 × 190 mm² with a 10 mm resolution. This shows that the pressure amplitude within this device is significant extending to zero-

to-peak amplitudes in excess of 400 kPa. This is significant as the threshold for inertial (or transient) cavitation can be estimated using standard literature models⁴² to be of the order of 129 kPa considering an operating ultrasonic frequency of 46 kHz in water under standard conditions. Clearly the acoustic pressure amplitude exceeds this value in multiple locations within the bath. However, it should be noted that it is necessary to have both a suitable bubble nuclei and sufficient acoustic pressure amplitude to produce inertial cavitation events. Figure 5b shows the results of scanning the ultrasonic bath over an identical range to that used for Figure 5a using the electrochemical erosion/corrosion approach, the opto-isolated system, and the MCA data analysis strategy. Figure 5b shows that there are indeed areas where inertial events (as determined by erosion/corrosion of the aluminum electrode) are detected and places where little or no erosion/corrosion events are detected. Over the range scanned up to 20 k events s⁻¹ were recorded in some locations. These locations also correlate well with the zones in the bath where significant pressure amplitudes were measured with the hydrophone (see Figure 5a). However, in some locations (for example point "A" on Figure 5b) no erosion/corrosion events were detected although the pressure amplitude was in excess of the inertial threshold. This illustrates an important point. Although it is necessary for the pressure amplitude to be in excess of the inertial threshold for erosion to be expected, it is also necessary to have an appropriate bubble population at that location. Point "A" shows that at this position and at the time of the experiment, these

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two criteria were not met. Figure 5 clearly shows the usefulness of this technique. It is possible by following this electrochemical strategy and by employing the opto-isolation to probe complex ultrasonic fields and to map them accurately to ascertain the actual erosive activity. However, because of the nature of many of these environments (an earthed metallic container), it is necessary to employ an isolation method (for example an opto-isolated system as employed here) for the electrochemical sensor to function adequately.

CONCLUSIONS

The weight loss calculated for a single current time transient is on the order of 46 fg or less. This is significant when compared to conventional cavitation weight loss measurements which are on the mg scale. The development of a simple opto-isolation method has been reported. This device in combination with a passivated electrode enables erosion/corrosion transients to be detected within an operating ultrasonic device. In this case the need to avoid electrical isolation from other electrode materials is avoided and "clean" transients recorded within the test environment employed. Mapping experiments have shown that it is possible to probe the erosive areas of a complex cavitation environment.

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