A self-contained, portable variable-pressure hydrostatic cell for use in low gauge pressure electromagnetic, ultrasonic, and photoacoustic studies

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A simple, manually adjustable hydrostatic cell for electromagnetic, ultrasonic, and photoacoustic studies in absolute pressure ranges from 40 kPa to 5.5 MPa is described. The cell consists of two 3-mm-thick quartz windows enclosing a 1.3-cm-diameter hole bored through a 2.54-cm-long cube block of stainless steel. Four 3-mm-diameter counterbored and taper-threaded holes on the cube walls provide minimally intrusive, chemically inert ports for temperature and acoustical monitoring. Pressure is easily varied within the cell by means of a stainless steel threaded shaft with an O-ring seal at the end, situated inside a matched internally threaded housing. An example of photoacoustic waveforms acquired under conditions of varying hydrostatic pressure is provided. © *1999 American Institute of Physics.* [S0034-6748(99)02201-7]

I. INTRODUCTION

Invasive monitoring of cardiac chamber pressures is a cornerstone of modern intensive care medicine. Monitoring, however, requires the insertion of a pressure sensor into the heart and the pulmonary veins. A recent, well-received study has raised questions as to whether the risk of monitoring outweighs the benefit of the data acquired.[1] As such, a search has begun for a new technology which may be used to monitor cardiac pressures noninvasively, that is, without direct physical contact between the monitoring sensor and the blood within the heart.[2] Additionally, gauge pressures on the order of kiloPascals or less are of great significance in processes as different as the systolic reduction of echogenicity of contrast enhancement in the left ventricle of the heart,[3] and the complex aggregation of human platelets observed *in vitro*.[4] The cell described below has been designed for precisely such research. Many previous articles have described the design challenges and ingenuity necessary for high pressure experimental apparatus.[5] However, the design of portable, convenient-to-operate apparatus useful in modest vacuum and lower and mid-(gauge) pressure ranges (from 40 kPa to 5.5 MPa absolute), with flexibility in probe and detection mechanisms (i.e., ultrasonics, lightwaves, microwaves) presents its own set of technical challenges.

II. DESCRIPTION OF APPARATUS

As Crippa so presciently states: ''Experience and serendipity are the two pillars of [photoacoustic] cell design.'' [6] Therefore, since the cell was to be initially used for photoacoustic work, it was modeled after the classic cell of Patel and Tam.[7] A sketch is shown in Fig. 1. The chamber body was bored out of a cube of type 303 stainless steel. The bore was 1.3 cm in diameter, and 2.54 cm in length. The surface of the bore was smoothed by the boring process but not further polished. The windows of the chamber were 3 mm thick, 25-mm-diameter annealed quartz with a coating to reduce reflection: neoprene O rings were used between the quartz window and the end surface of the chamber. The liquids in the chamber were consequently exposed to stainless steel, coated quartz, Teflon® (due to the flexural supporting members to be discussed later), neoprene, and brass. The presence of brass was due to the pressure gauge—clearly a stainless steel gauge could have also been used.

Four 3-mm-diameter holes passed through each of the remaining four faces of the cube into the cell chamber. The holes were counterbored to a width of 8 mm and taper-threaded externally. The acoustical, temperature, and pressure sensors as well as the pressure-varying device (the ''piston'') were attached to the cell body using adapters that mated the devices to the threaded holes. The piston itself consisted of a stainless steel threaded shaft with an O-ring seal at the end: the shaft was situated inside a matched internally threaded housing. Due to the near incompressibility of liquids, relatively large pressures could be applied to the chamber with only a few turns of the handle of the piston. Although the cell was not designed for high pressures, a test run revealed that the cell retained pressures up to 5.5 MPa, when the thin cell windows cracked. Clearly, if higher pressure ranges were desired, these could be achievable with thicker windows.

Air bubbles were removed from the chamber by setting the chamber on its side, removing one of the chamber windows, and screwing the piston down into (towards) the chamber as far as possible. A long hypodermic needle was then used to inject liquid into the inlets of the pressure gauge and the piston (thereby removing pockets of air which would otherwise have remained if liquid had simply been poured into the chamber) and to fill the chamber itself with liquid. Once the chamber was filled with liquid, the piston was

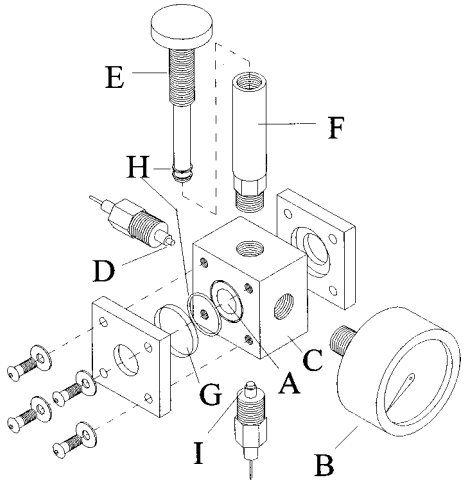

FIG. 1. Photoacoustic pressurization cell (A), also showing pressure gauge (B), stainless steel housing (C), thermistor (D), piston (E), piston bore (F), quartz windows (G), gaskets (H), and hydrophone (I).

gradually drawn to its extended position while simultaneously adding more liquid to replace that which was pulled into the piston bore. Finally, the window was replaced while carefully ensuring that no air bubbles slipped underneath it before tightening.

Vacuum pressures could be achieved in the chamber simply by replacing the window without drawing the piston to its extended position. Once the window was replaced, the piston could be drawn back out, sucking liquid out of the chamber as it moved and leaving the remaining liquid at a lower pressure. An estimated 40 kPa absolute pressure was achievable in this fashion.

The hydrostatic pressure gauge utilized for experimental purposes was a simple diaphragm-type dial gauge capable of indicating pressures between 0 to 2 MPa above atmospheric. The use of a Bourdon-tube gauge would have been inappropriate, as the large air pocket in the tube of the gauge would have been difficult to eliminate. In the final tests to determine pressure ranges of the cell itself, a 0–6 MPa gauge was used.

As with the pressure gauge, ancillary instrumentation for the cell was dictated by the requirements of our experiments. The thermistor was Teflon®-coated with ± 0.1 °C accuracy. The hydrophone was a spot-poled reflector type hydrophone manufactured by Specialty Engineering Associates of Soquel, CA. This hydrophone has a flat, circular tip about 2.4 mm in diameter. It was not a pressure-compensated device–pressure compensation would be recommended for work at the mid- and higher-pressure ranges of the cell.

The front surface of the hydrophone extended approximately 5 mm into the chamber. The hydrophone was housed within a Teflon® tube of approximately 6-mm-outside diameter, which was slightly larger than that of the hydrophone. The hydrophone was gently inserted into the tube and a stainless steel Swagelok ferrule was placed over the assembly. The ferrule was tightened by a nut as the assembly was threaded into the photoacoustic chamber. Ferrules of more elastic material, such as PEEK, KEL-F® [PEEK and KEL-F® are both different types of high performance plastics. PEEK stands for polyetheretherketone, while KEL-F® is a fluorochemical product also known as CTFE (chlorotrifluorethylene)] or Teflon® might have deformed as the nut was tightened, which would have meant that the ferrule would have had to have been replaced each time the hydrophone was removed from the chamber. However, such materials might be considered in future work to reduce the risk of damage due to overtightening. A number of other mounting mechanisms were tried, as for instance, an o-ring type assembly, however, these mounts eliminated the signal from the hydrophone as soon as any clamping took place (i.e., as soon as the hydrophone was tightened into the holder, but before any pressure was applied to the cell). It appears that the long Teflon® tube applied smaller and more evenly distributed forces per unit area than the other types of mounts that were tried. To avoid excessive crimping on the fine wires of the thermistor, a Teflon® tube configuration similar to that of the hydrophone was used.

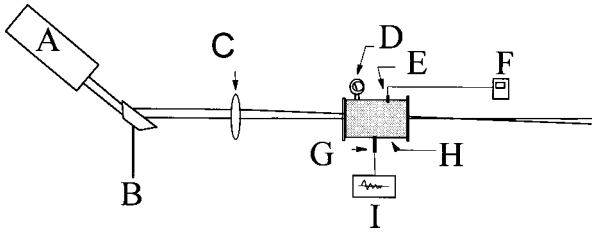

FIG. 2. Experimental setup: Nd:Yag laser (A), Pellin–Broca prism (B), focusing lens (C), hydrostatic pressure gauge (D), thermocouple (E), ohmmeter (F), hydrophone (G), photoacoustic chamber (H), 1 GHz digital storage oscilloscope (I).

The tip of the hydrophone was extraordinarily sensitive to damage: even threading a simple o ring onto the hydrophone could damage it beyond repair. A 3-mm-thick Teflon® washer, slightly larger in interior diameter than the hydrophone, and slightly smaller in outer diameter than the 8-mm-counterbored hole, was inserted at the sides of the hydrophone hole in the photoacoustic chamber: this insert proved to be very helpful in eliminating scratches and other damage to the hydrophone as it was pushed into the chamber. It should be noted that damage during mounting can occur which will result in the hydrophone showing a normal signal at atmospheric pressure, and no signal at gauge pressures of only a few kPa.

A problem with the hydrophone mount was that the hydrophone could not be removed without the possibility of damage. This meant that it was very difficult to try to completely remove all trace of a solution from the chamber, since even after careful rinsing, some portion of the solution would remain in the cracks between the hydrophone, the Teflon® tube, and the photoacoustic chamber wall. Most of this contamination could be removed, however, by rinsing the chamber and then sucking the contamination out of cracks and crevices through the application of low pressure (as previously described) two or three times.

III. APPLICATIONS

The cell was initially designed and put to use in studying the effect of pressure on the photoacoustic signals of various solutions. The experimental setup is shown in Fig. 2. The

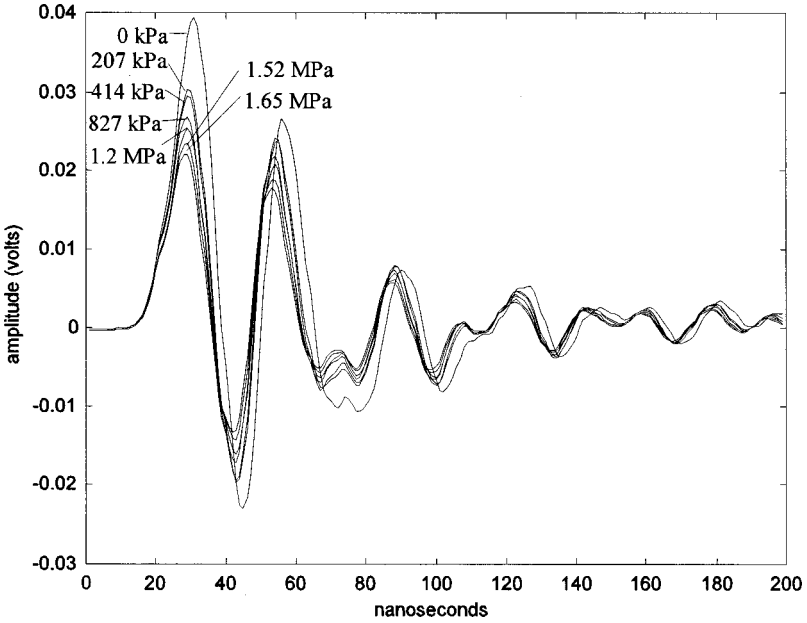

FIG. 3. Photoacoustic waveforms of tris(2,2′-bypyridine) ruthenium (II) chloride (126 mJ/pulse; beam diameter=0.42 cm. o.d. 0.803, 532 nm, 9:1 acetonitrile: water). Gauge pressures as indicated for the various waveforms. Waveforms are each the average of approximately 200 acquisitions.

laser used to generate the photoacoustic signal was a diffraction-coupled *Q*-switched Nd:YAG laser with nominal 10 ns pulses operating at 10 Hz. Experiments were conducted at wavelengths of 532 and 355 nm. Laser energy during all runs was standardized at the relatively high value of 126 mJ per pulse (estimated within the photoacoustic chamber). The laser beam was sent through a Pellin–Broca prism to separate the wavelengths. A focusing lens was placed between the prism and the chamber: the average beamwidth through the liquid in the chamber was varied from 0.36 to 0.6 cm.

Photoacoustic signals from the hydrophone were acquired by a LeCroy 1 GHz digital storage oscilloscope (up to 1 sample point each nanosecond on single shot mode). Signals could be averaged over up to 1000 waveform acquisitions. The waveform acquisition triggered by the rising amplitude of the waveform itself; no external triggering was supplied. Waveform data was stored on 3.5-in.-floppy disks in ASCII compatible with MATLAB™ via an onboard high density 3.5-in-floppy disk drive (DOS format). The stored waveforms were imported into MATLAB™ and thereby further analyzed. Results from an experiment for a solution of tris(2,2′-bypyridine) ruthenium (II) chloride are shown in Fig. 3. Here the magnitude of the amplitude of the first, compressive, portion of the waveform is directly proportional to the amount of ''prompt'' (i.e., below the experimental integration time of the hydrophone) heat released into the solution. These results must be evaluated with caution, as some hysteresis was experienced in the hydrophone output.

Future work will involve further photoacoustic experiments with various hydrophones, as well as experiments involving ultrasound and microwave signals.

ACKNOWLEDGMENTS

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