

**“Enhanced Wellbore Stabilization and Reservoir Productivity with  
Aphron Drilling Fluid Technology”**

**QUARTERLY PROGRESS REPORT**

**January 1 – March 31, 2004**

**by**

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## ABSTRACT

During this second Quarter of the Project, the first four tasks of Phase I – all focusing on the behavior of aphrons -- were continued: (a) Aphron Visualization – evaluate and utilize various methods of monitoring and measuring aphron size distribution at elevated pressure; (b) Fluid Density – investigate the effects of pressure, temperature and chemical composition on the survivability of aphrons; (c) Aphron Air Diffusivity – determine the rate of loss of air from aphrons during pressurization; and (d) Pressure Transmissibility – determine whether aphron bridges created in fractures and pore throats reduce fracture propagation.

The project team expanded the laboratory facilities and purchased a high-pressure system to measure bubble size distribution, a dissolved oxygen (DO) probe and computers for data acquisition. Although MASI Technologies LLC is not explicitly ISO-certified, all procedures are being documented in a manner commensurate with ISO 9001 certification, including equipment inventory and calibration, data gathering and reporting, chemical inventory and supplier data base, waste management procedures and emergency response plan.

Several opportunities presented themselves to share the latest aphron drilling fluid technology with potential clients, including presentation of papers and working exhibit booths at the IADC/SPE Drilling Conference and the SPE Coiled Tubing Conference & Exhibition. In addition, a brief trip to the Formation Damage Symposium resulted in contacts for possible collaboration with ActiSystems, the University of Alberta and TUDRP/ACTS at the University of Tulsa.

Preliminary results indicate that the Aphron Visualization and Pressure Transmissibility tasks should be completed on time. Although the Aphron Air Diffusivity task has been impeded by the lack of a suitable DO probe, it is hoped to be completed on time, too. The Fluid Density task, on the other hand, has had significant delays caused by faulty equipment and will likely require an additional month of work. Meanwhile, an assessment of potential methodologies for the Aphron Hydrophobicity project has been initiated and is now focused on measuring wettability of the aphron surface rather than interfacial tension.

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## INTRODUCTION

Aphron drilling fluids were developed as a more economical and safer alternative to underbalanced drilling. Air is entrained during normal mixing operations – obviating the need for gas injectors and pumps – at such a low concentration that, under downhole conditions, it does not affect the fluid density significantly. Aphron drilling fluids are thought to seal permeable zones by virtue of their very high low-shear rheology and the unique bridging capability of specially designed microbubbles, or “aphrons”.<sup>1</sup> An aphron is hypothesized to consist of a core of air enveloped by a protective shell composed of multiple layers of surfactants and polymers.<sup>2</sup> The outer part of the structure consists of a surfactant bi-layer that renders the aphron water-wet and, therefore, compatible with the continuous aqueous phase. However, the outermost surfactant layer in the bi-layer may be shed when aphrons are forced together in a pore throat or near a fracture tip.<sup>1</sup> Under these conditions, aphrons could acquire some oil-wetting character that would permit them to agglomerate without coalescing and seal off the opening without wetting the walls.

Neither the structure of the aphron nor its physicochemical properties have been validated sufficiently for the technology to be widely accepted. Acceptance of this novel technology and the consequent reduction in drilling costs would be facilitated greatly by a systematic and thorough evaluation of aphron drilling fluids to gain some understanding of the structure of aphrons and how aphron drilling fluids behave downhole.

The objectives of this project are threefold: (a) develop a comprehensive understanding of how aphrons behave at elevated pressures and temperatures; (b) measure the ability of aphron drilling fluids to seal permeable and fractured formations under simulated downhole conditions; and (c) determine the role played by each component of the drilling fluid.

The Project is divided into two phases. Phase I is focused on developing evidence for the ways in which aphrons behave differently from ordinary surfactant-stabilized bubbles, particularly how they seal permeable and fractured formations during drilling operations. Key properties to be investigated include the effects of pressure on bubble size, the oil-wetting/water-wetting nature of the aphron surface under dynamic conditions, and the nature of aphron seals in fractures

and pore networks. Initial sealing and formation damage tests also will be carried out, using lab-scale apparatus designed to simulate permeable and fractured environments. Phase II focuses on sealing and formation damage testing of aphon drilling fluids, including scale-up tests under simulated downhole drilling conditions, so as to furnish irrefutable evidence for the validity of this technology and provide field-usuable data.

The current schedule of tasks is provided in Figure 1.

Task	2003			2004			2005		
	4th Q	1st Q	2nd Q	3rd Q	4th Q	1st Q	2nd Q	3rd Q	
I. Physical Characterization of Aphrons									
<b>1a. Aphon Visualization</b>	X	X	X	X					
<b>1b. Fluid Density</b>	X	X	X*						
<b>1c. Aphon Air Diffusivity</b>	X	X	X						
2a. In Situ Visualization			X	X					
<b>2b. Pressure Transmissibility</b>	X	X	X						
2c. Aphon Shell Hydrophobicity			X	X					
3a. Sealing of Permeable Media			X	X					
3b. Sealing of Fractured Media			X	X					
II. Characterization of Aphon Drilling Fluids									
1a. Lab Tests Leak-Off/Return Perm					X	X	X		
1b. Field-Sim Tests Leak-Off/Return Perm							X	X	
2a. Flow Sim through & Sealing Fractures					X	X	X	X	
2b. Fracture Re-Opening Tests						X	X	X	

\* Extension Proposed April 1, 2004

**Figure 1. Schedule of Tasks to be Performed in Aphon Drilling Fluid Project**

The tasks highlighted in red were begun during the 4<sup>th</sup> Quarter of 2003. These have the following objectives:

- 1a. Aphon Visualization - Evaluate the Acoustic Bubble Spectrometer (ABS) procedure to determine if the method is applicable for future measurements of bubble size distribution (BSD) in a pressurized environment. Determine the effects of pressure, temperature and chemical composition on the BSD of aphon drilling fluids.
- 1b. Fluid Density - Investigate the effects of pressure, temperature and chemical composition on the density of aphon drilling fluids.

- 1c. Aphron Air Diffusivity - Determine the effects of pressure, temperature and chemical composition of aphron drilling fluids on the rate of loss of air from aphrons and, hence, the importance of air loss on the survivability of aphrons.
- 2b. Pressure Transmissibility - Investigate the rate and magnitude of pressure transmission in simulated fractures and in permeable rock bridged with aphrons to test the hypothesis that aphron networks can reduce mud loss via reduction of pressure transmissibility.

## EXECUTIVE SUMMARY

Four tasks were begun during this Quarter. All of these focus on the behavior of aphrons: (a) Aphron Visualization – evaluation of potential methods for measurement of bubble size distribution (BSD), especially Acoustic Bubble Spectroscopy (ABS), in aphon drilling fluids at elevated pressure; (b) Fluid Density – investigation of the effects of pressure, temperature and chemical composition on the survivability of aphrons; (c) Aphron Air Diffusivity – determination of the rate of loss of air from aphrons during pressurization; and (d) Pressure Transmissibility – evaluation of the ability of aphon aggregates formed in fractures and pore networks to reduce fracture propagation.

The project team expanded the laboratory facilities and purchased two computers for processing data from the syringe pumps of the fluid density experiments, the dissolved oxygen probe for the aphon diffusivity tests, transducers on the pressure transmissibility apparatus and a bank of four concentric cylinder viscometers.

The Aphron Visualization task appears to be on schedule, despite delays caused by ABS software problems, and should be completed on time (9/30/04). Results to date indicate that it may be possible to correlate BSD derived from ABS with BSD determined via conventional laser light scattering. In addition, a locally available Environmental Scanning Electron Microscope has been checked out and appears to be suitable for *in situ* visualization of the sealing of pore networks.

The Fluid Density task, on the other hand, has had some fundamental theoretical and equipment problems; completion of this task is expected to be delayed about six weeks (to 5/15/04). The key changes in the project include (a) use of pressure cycling to examine the hysteresis in the fluid density, rather than the density itself, and (b) honing and polishing of the interior of the pressure vessel and replacement of the piston with a material that provides a tighter seal.

The Aphron Air Diffusivity task appears to be on schedule (completion date 6/30/04), though it, too, has experienced equipment problems. A fluorescence probe has been evaluated for its ability to monitor the rate of change in the concentration of dissolved oxygen, hence the rate of loss of air from aphrons. Unfortunately, the protective coating on the probe suffers from delamination in the drilling fluid, and the response of the probe is quite sensitive to fluid viscosity. A solution is being sought.

Lastly, the Pressure Transmissibility task is expected to be completed on time (6/30/04), though there have been significant questions about the ability to create aphon aggregates in the experimental apparatus. The most recent apparatus consists of a 6 m (20 ft) straight piece of 0.64 cm (1/4") tubing with a gravity-feed pressure-assisted filling system. Rate of pressure transmission and steady-state pressure drop along the length of the tube are monitored.

Preliminary work has also begun on tasks that will be initiated in the 3<sup>rd</sup> Quarter of this project, namely Aphron Hydrophobicity, *In Situ* Aphron Visualization and Sealing of Permeable and

Fractured Media. Initial literature searches and assessment of appropriate test methods were completed. For the Aphron Hydrophobicity task, the focus has shifted to contact angle goniometry, rather than interfacial tension as a means to quantify adhesion of aphrons on fracture and pore surfaces. For the In Situ Visualization task, a high-pressure high-temperature circulating system has been built. However, the cell has had to be re-designed and will be built locally to enable experimentation at the target conditions of 20.7 MPa (3,000 psi) and 121 °C (250 °F). Finally, for the Sealing tests, a triaxial loading core leak-off tester has been ordered.

Although MASI Technologies LLC is not explicitly ISO-certified, all procedures are being documented in manner commensurate with ISO 9001 certification, including equipment inventory and calibration, data gathering and reporting, chemical inventory and supplier data base, waste management procedures and emergency response plan.

Two meetings were held with DOE representatives during this Quarter:

- Project Kick-Off Meeting with NETL DOE Project Team in Morgantown , W. Va. on Jan 23<sup>rd</sup> , followed by a visit to Dynaflow, Inc. (supplier of HTHP ABS System) in Jessup, Md. on Jan 26<sup>th</sup> .
- Informal technical update and lab tour with Gary Covatch and John Rogers Feb. 18 at the M-I Technology Center and Applied Engineering Lab, Houston, TX, where the DOE Project Team is temporarily ensconced.

Several opportunities presented themselves to share the latest aphron drilling fluid technology with potential clients. These included

- A brief trip to Lafayette, LA Feb. 19 & 20 to meet with attendees at Formation Damage Symposium, including Sandra Cobianco of ENI Tecnologie (Milano, Italy) and Dr. Ergun Kuru of the University of Alberta (Edmonton, Canada) re formation damage and extensional viscosity of aphron drilling fluids. Also met with Jack Cowan of ActiSystems Inc. (partner in MASI Technologies LLC) to return that company's ABS System and discuss collaborative efforts. Finally, a brief meeting was held with Dr. Nick Takach of the University of Tulsa to discuss flow of foams and energized fluids; Dr. Takach invited the team to participate in the TUDRP/ACTS (Tulsa University Drilling Research Program/ Advanced Cuttings Transport Study) annual meeting in May.
- Presentation of paper # 87134, "Alternative Aphron-Based Drilling Fluid," at IADC/SPE Drilling Conference in Dallas, TX, March 2-4.
- Exhibit Booth duty at SPE Coiled Tubing Conference & Exhibition, The Woodlands, TX, March 23, 24, to meet clients and ascertain what they consider the technology gaps in aphron drilling fluid technology,.

# EXPERIMENTAL APPROACH

The methodologies used for the four current tasks are detailed below:

## Aphron Visualization

Formulate a mud that is representative of the APHRON ICS mud system, yet transparent to light:

- FloVis Plus 8.6 g/L (3 lb/bbl)
- 50% NaOH to pH 10
- Blue Streak 2.9 g/L (1 lb/bbl)
- EMI-779 1.4 g/L (0.5 lb/bbl)
- EMI-780 1.4 g/L (0.5 lb/bbl)

Measure BSD using photomicrography; acoustic bubble spectroscopy, or ABS (see Figure 2); and laser light scattering (Figure 3). Compare the three methods using the following fluids:

- Transparent Aphron Mud Formulation without entrained air
- Transparent Aphron Mud Formulation with varying amounts of entrained air and varying shear history.

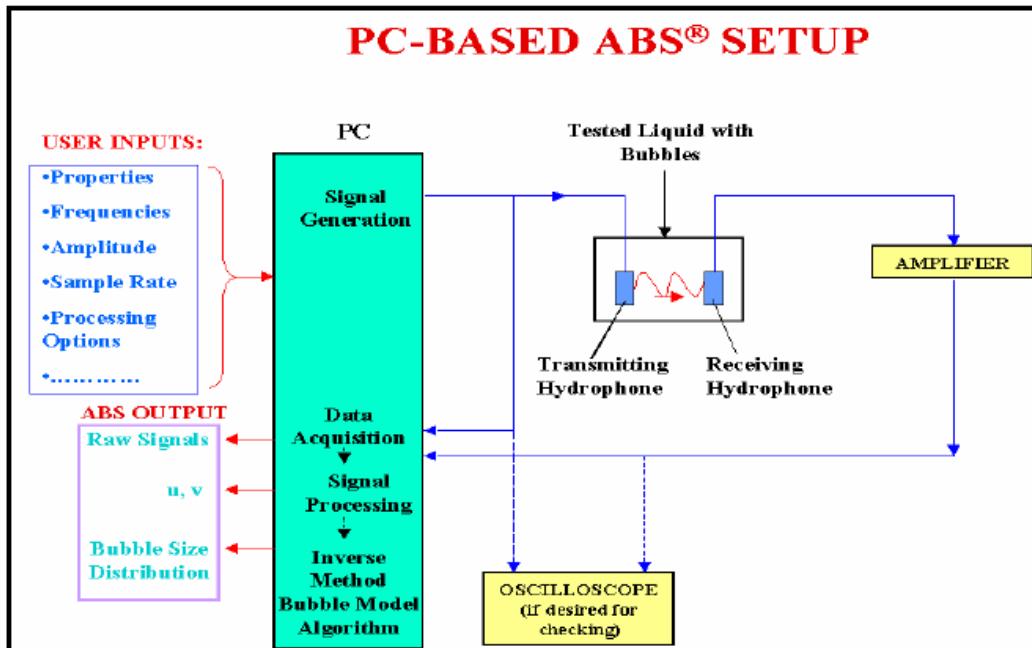


Figure 2. Configuration of Dynaflow Acoustic Bubble Spectrometer<sup>3</sup>



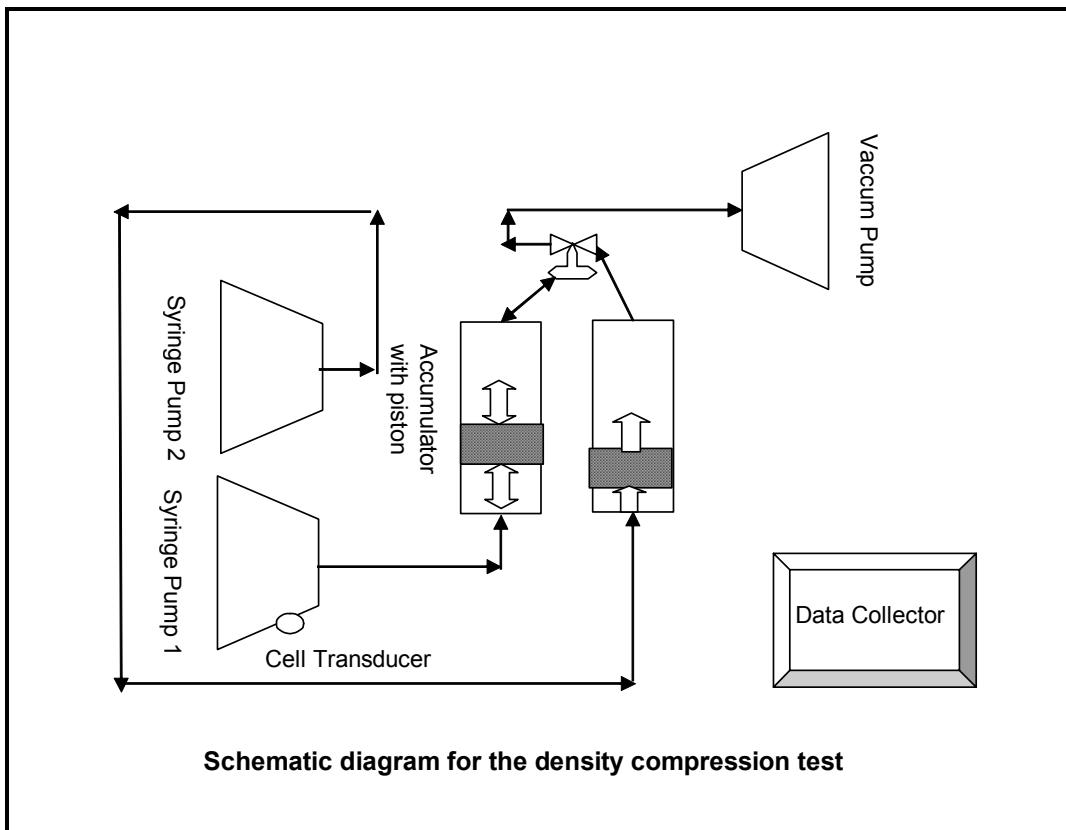
**Figure 3. Coulter Laser Light Scattering System**

### Fluid Density

Determine survivability of aphrons by measuring the effect of pressure on fluid volume and density, from which the concentration of undissolved air can be determined.

Carry out initial tests with a standard aphon-laden drilling fluid containing variable amounts of air, and generate density vs pressure curves over a range of fluid temperatures. Later, examine the effects of different surfactants and polymers on the survivability of the aphrons.

The apparatus used to carry out these tests has been modified numerous times to minimize accidental entrainment of air and facilitate carrying out the ramped pressure tests. A schematic of the apparatus is shown below in Figure 4:



**Figure 4. Latest Design of Apparatus for Fluid Density Measurements**

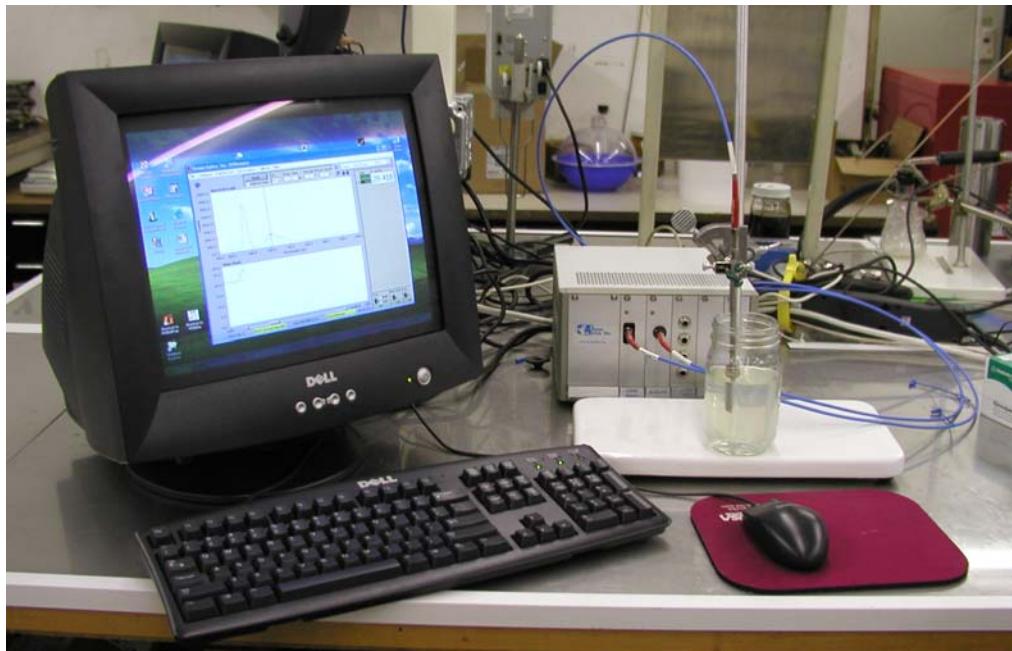
### Aphron Air Diffusivity

To measure the rate of loss of air from aphrons, examine the possibility of measuring the rate of increase of Dissolved Oxygen (DO) in the surrounding aqueous medium. Determine the principle underlying operation of various DO probes, their accuracy and reproducibility, methods of using them, and their limitations, e.g. effect of pressure.

Construct a vessel that can accommodate a suitable DO probe and enable measurements of DO at elevated pressures and temperatures.

Run experiments to determine the effects of pressure, temperature and chemical composition on the rate of build-up of dissolved air. If possible, run complementary tests to measure the corresponding change in bubble size that can be correlated with the rate of increase in the concentration of dissolved air in the mud.

A FOXY DO probe has been obtained from OceanOptics for evaluation. Figure 5 shows the system configuration currently being employed:

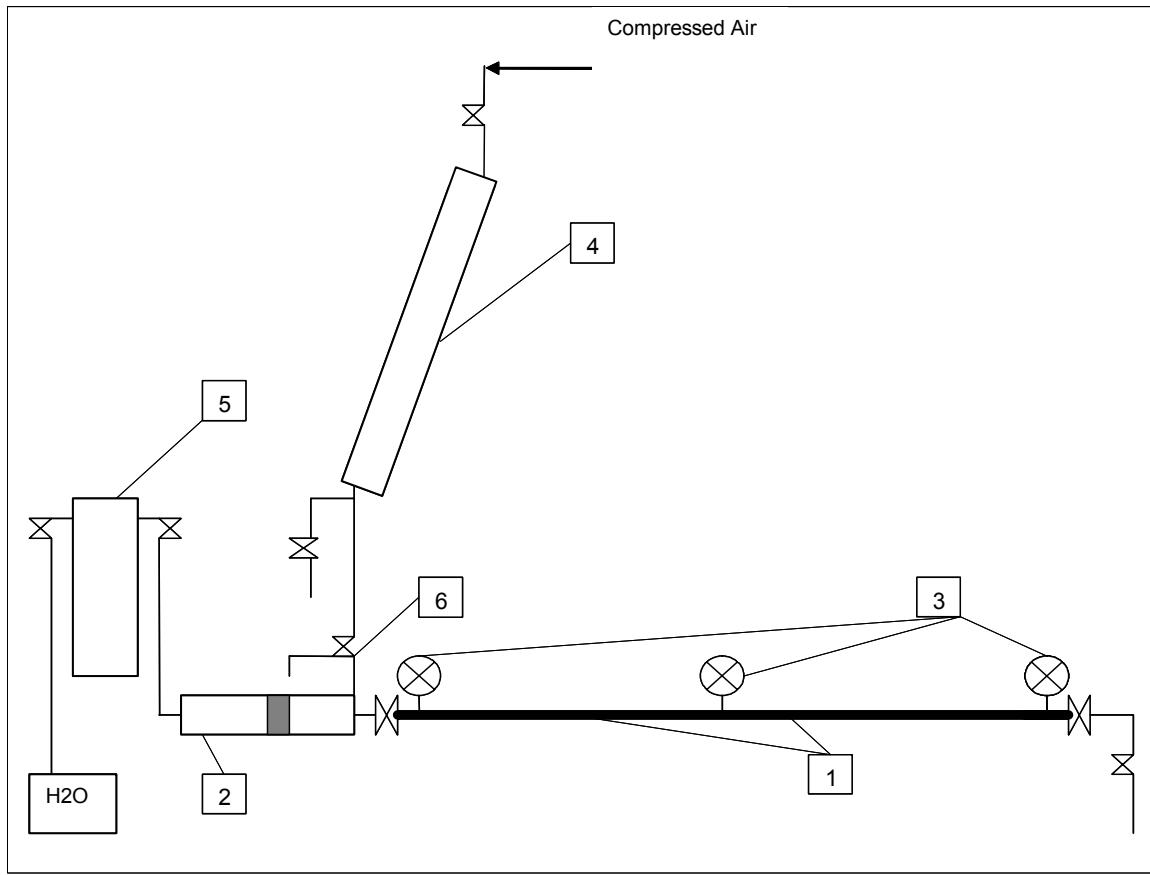


**Figure 5. FOXY Dissolved Oxygen System for Air Diffusivity Tests**

### Pressure Transmissibility

- Design and construct an appropriate system to monitor the rate, as well as the amplitude, of pressure transmission through aphron drilling fluids and aphron “bridges” in a simulated fracture and permeable rock. Begin by simulating a fracture using an existing 76-m (250 ft) length of 0.64-cm (1/4") OD stainless steel tubing fitted with three pressure transducers along its length.
- When a suitable system has been constructed, measure steady-state pressure drop and the rate of pressure transmission through standard APHRON ICS fluid as a function of the concentration of air and aphron-stabilizing components.

The apparatus currently being used is illustrated in Figure 6.



**Figure 6. Layout of Pressure Transmissibility Apparatus**

## RESULTS AND DISCUSSION

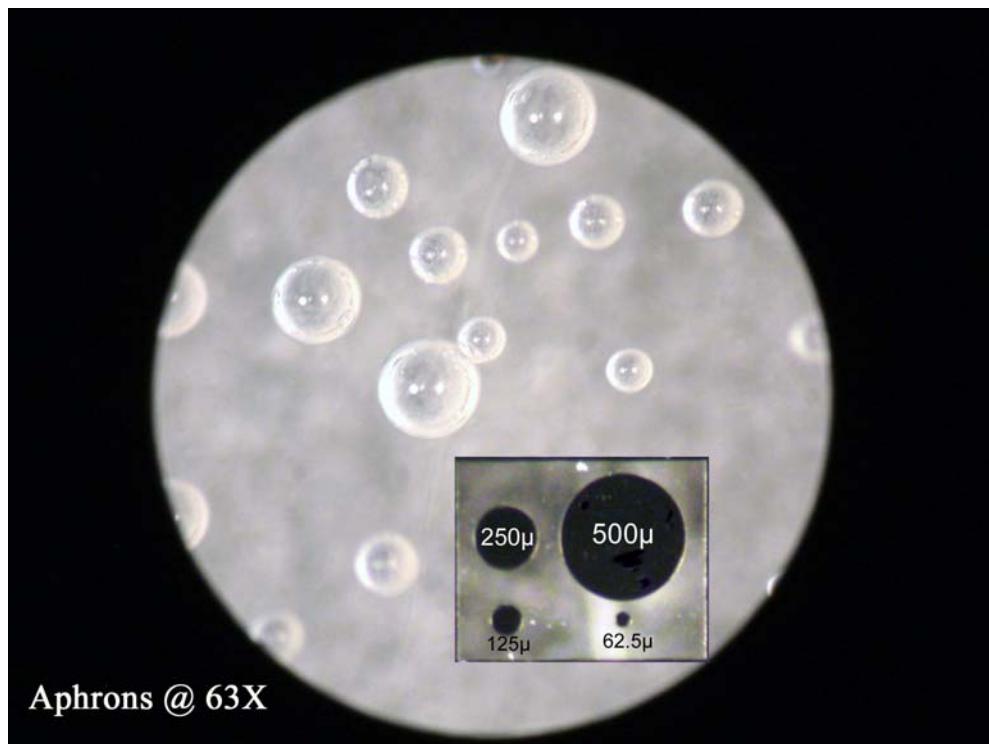
### Aphron Visualization

The SCSI board checked by Dynaflow was damaged during its return. It has now been repaired by the board manufacturer. Although the ABS computer is now working, the ABS software remains unstable.

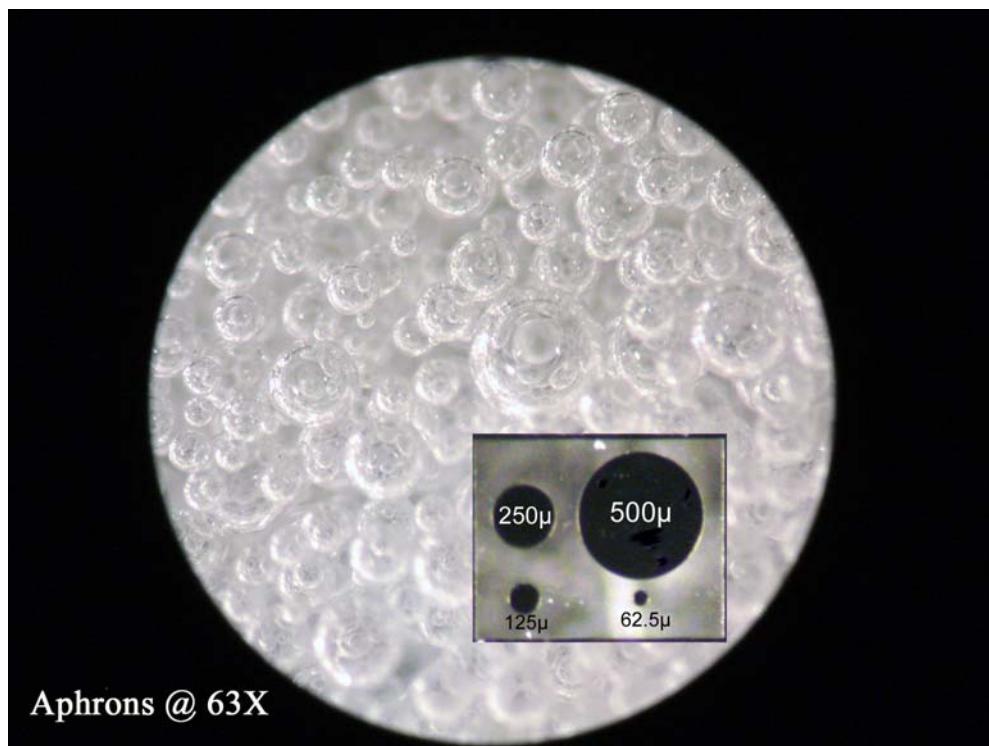
The HTHP ABS System being constructed by Dynaflow, Inc. has had a number of problems. The two-phase motor for the circulating pump is being replaced with a single-phase motor to accommodate our electrical capabilities. The safety of the ABS Cell has been brought into question; a local engineering firm was asked to evaluate the design of the Cell, and it was concluded that the Cell, particularly the end-caps, is not safe to use at the specified working pressure of 20.7 MPa (3,000 psi) and 121 °C (250 °F). Furthermore, it is likely that cell will not be able to seal at that pressure (at least not repeatedly), nor does the cell lend itself to being dismantled (use of pipe threads for such a large vessel, along with the same metallurgy for the caps and body will cause galling). At this juncture, it is not known when the HTHP ABS System will be ready.

Some photomicrographs of aphrons in the transparent mud formulation were obtained, again at ambient temperature and pressure. A Coulter Laser Light Scattering System usually employed for particle size distribution of weighting materials is being examined to determine whether it can yield accurate visual bubble size distributions (BSD) of the aphrons; initial feedback from the M-I Analytical Dept is somewhat encouraging, though an arrangement involving a high-pressure polycarbonate cell with an optical imaging system may be more satisfactory. Photomicrographs of aphrons from a diluted and a conventional APHRON ICS mud are shown in Figures 7 and 8.

Information gathered on the viability of using an Environmental Scanning Electron Microscope indicates it may be useful for observing flow and sealing of pore networks by aphrons.



**Figure 7. Aphrons in a Diluted APHRON ICS Mud Sample**



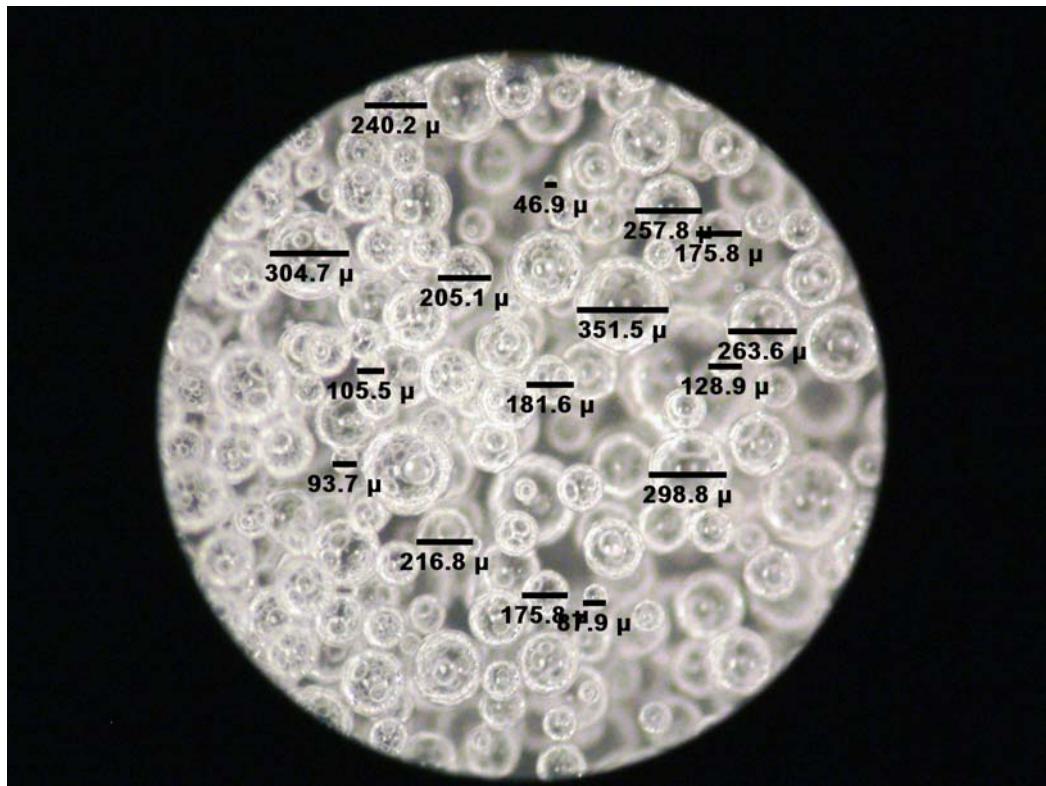
**Figure 8. Aphrons in a Conventional APHRON ICS Mud Sample**

We received a much more stable version of the Acoustic Bubble Spectrometer(ABS) software from Dynaflow, Inc. and began performing ABS tests. ABS tests were run on  $\sim 500\mu\text{m}$  glass beads and compared to photomicrographs of the beads. The ABS results indicated that the beads were of a size  $\leq 200\mu\text{m}$  and of various sizes. However, only  $500\mu\text{m}$  glass beads were observed in the photomicrographs. Thus, the ABS generates a PSD that is skewed to very low values; it remains to be seen whether the same is true for bubbles.

In addition, we brought to our lab another ABS unit and an experimenter from ActiSystems, Inc. and ran both units side by side. The results between the two units run by different operators were almost identical. This experiment tested the stability of the software and confirmed that we were performing the tests properly.

As we continue to gather data and compare it with the photomicrographs and laser light scattering PSD (particle size distribution), we will determine if we can use the ABS data directly or whether the data will have to be normalized to accurately reflect the bubble size distribution. To this end, we are using software for analysis of the photomicrography data that applies the diameter of the bubble directly to the image. An example is shown in Figure 9.

ABS experiments were performed to determine the effect of varying the sound frequency range picked up by the instrument on the BSD. Generally, low frequencies are more sensitive to large diameter bubbles, whereas high frequencies are more sensitive to small diameter bubbles. However, no significant difference was observed when the window was varied from as wide as 50 – 250 kHz to as narrow as 140 – 150 kHz. Nevertheless, the frequency window was left as wide as possible to ensure maximum sensitivity to all bubble sizes. Additional tests were performed to determine if there was any effect of varying the sound spectrum analysis to adjust the bubble size range. Here again, the same pattern was observed whether using 0 – 1000 $\mu\text{m}$  or 0 – 500 $\mu\text{m}$ .



**Figure 9. Image Analysis Software for Photomicrographic BSD Measurements**

All of these BSD's with the ABS were compared to BSD's of the same samples determined via Photomicrographic analysis and Laser Light Scattering (LLS). The ABS results appear to be compressed and shifted to bubble sizes an order of magnitude lower than those measured via photomicrography or LLS. Greater sensitivity and perhaps better agreement may result when the 1" x 1" hydrophones are replaced with 3" x 3" hydrophones.

Also, we have been investigating the following instruments to determine their usefulness as analytical methods for APHRON ICS Muds:

- JM Carty Flow-Through Cell: Ability to carry out photomicrographic image analysis of fluids at elevated temperatures and pressures.
- Capillary Suction Time Tester: May be applicable as an alternative to the triaxial core leak-off apparatus and for studying extensional viscosity of Aphrons.

The HTHP ABS apparatus was received from Dynaflow, Inc.; however, safety and handling problems with the cell require reconstruction of the cell. A new cell has now been designed and will be built shortly.

## Fluid Density

The apparatus was modified to reduce error introduced by air in the plumbing system, as well as to better fit the new lab setup at the Applied Engineering Lab.

The new system was constructed and calibrated, and a few tests were performed with water at pressures up to 6.9 MPa (1000 psia). The system, at best, still shows 1 to 2% undissolved air. This may not be acceptable. The original intent of this project was to determine if aphrons can survive pressurization to 1000+ psia. For a mud sample of 1000 mL which contains 15% air (aphrons), or 150 mL air, pressurization itself (with no loss of air to the surrounding mud) would reduce the volume of air to 2.25 mL (0.225%). The air that is “lost” is assumed to leak through the aphon shell and dissolve in the mud matrix. Thus, the experimental apparatus must be able to generate distinguishable results between the case where no air is lost vs all air is lost, i.e.  $\Delta V_0 = 147.8$  mL (no loss of air) vs  $\Delta V_{100} = 150$  mL (all air is lost). That should be feasible. However, the air trapped in the plumbing at the beginning of a test would generate a  $\Delta V$  of 10 to 20 mL. Thus,  $\Delta V_0^{\text{Corr}} = 157.8$  to 167.8 mL and  $\Delta V_{100}^{\text{Corr}} = 160$  to 170 mL. Since the uncertainty in the trapped air volume ( $\sim 10$  mL) is more than four times the volume difference we hope to measure ( $\Delta V_{100} - \Delta V_0 = 2.25$  mL), these tests must be modified.

Some preliminary research was carried out on the next project, “Aphon Shell Hydrophobicity,” which will begin at the conclusion of this project.

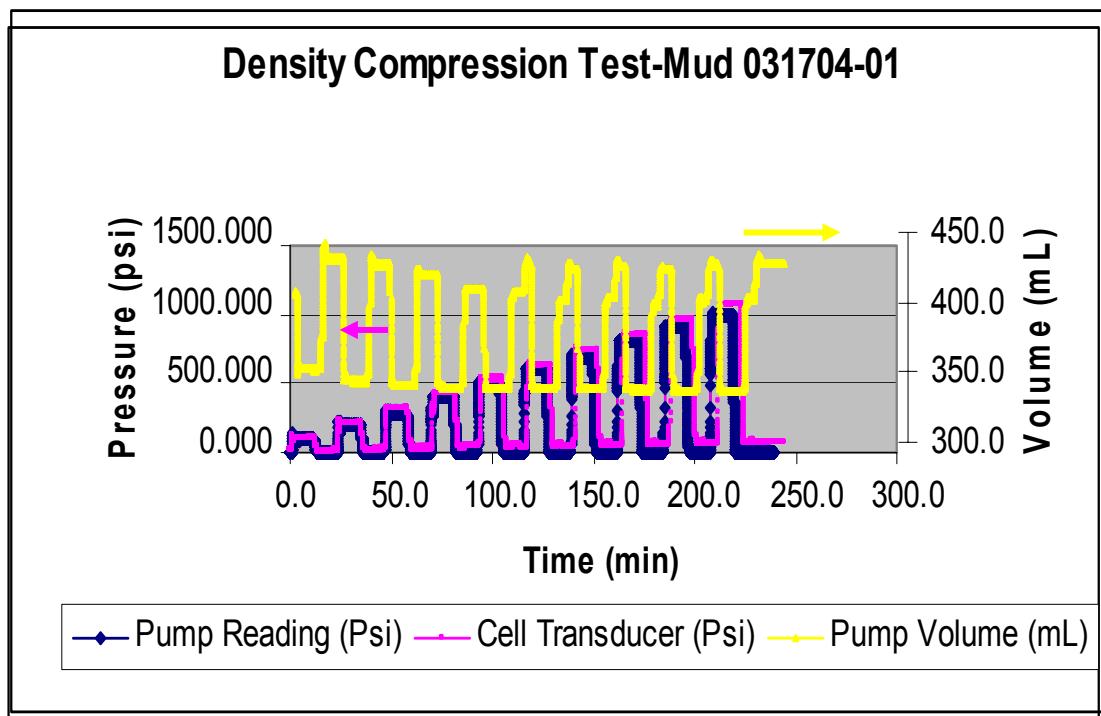
As mentioned in the last month’s report, analysis of the results is focused on hysteresis effects during pressurization/depressurization cycles. The mud used was the Super Enhanced Aphronics mud (containing both primary and secondary aphon stabilizers) with about 30% by volume air.

The pressure was varied in cycles, first increasing to the desired pressure (0.69 MPa or 100 psia, 1.38 MPa or 200 psia and so on up to 6.9 MPa or 1000 psia, in increments of 100 psi) over a period of 1.5 min and then decreasing it for 1.5 min down to 0.069 MPa (10 psia). We noticed that for each cycle, during the depressurizing step the air that was first compressed during the pres-

surizing step was not all coming back. We thought that this effect was probably due to the short time between the steps, so a 10-min delay was added between the steps (see Figure below).

At that point we saw something new in the behavior of the fluid. Most of the air (aphrons + air that escaped from the mud) was recovered at the end of each cycle, and eventually all of it at the end of the test. But the problem this time was the erratic nature of the volume readings (and to some extent to the pressure readings). We are not sure what is causing this. One possibility is stick/slip of the piston in the cell. To test this hypothesis, the cells have been returned to the manufacturer to have the barrels honed and the piston and o-ring seal replaced for a smoother movement and tighter fit. Meanwhile, more literature research is being done in the hope of gaining additional insight into this behavior of the mud.

A sample of the PVT data collected from the test of the Super Enhanced (SE) Aphronics mud with air at room temperature is shown in Figure 10:



**Figure 10. PVT Test of an SE APHRON ICS Mud**

## Aphron Air Diffusivity

The effect of viscosity on response time of conventional DO probes continued to be examined using samples of air-free AphronICS mud vs water to determine how to analyze the diffusivity data from the probes. An apparatus is being designed that will generate high-shear flow at the sensor, thus reducing the viscosity of the highly shear-thinning drilling fluid and decreasing its effect on the measurement of diffusivity through the aphron shell.

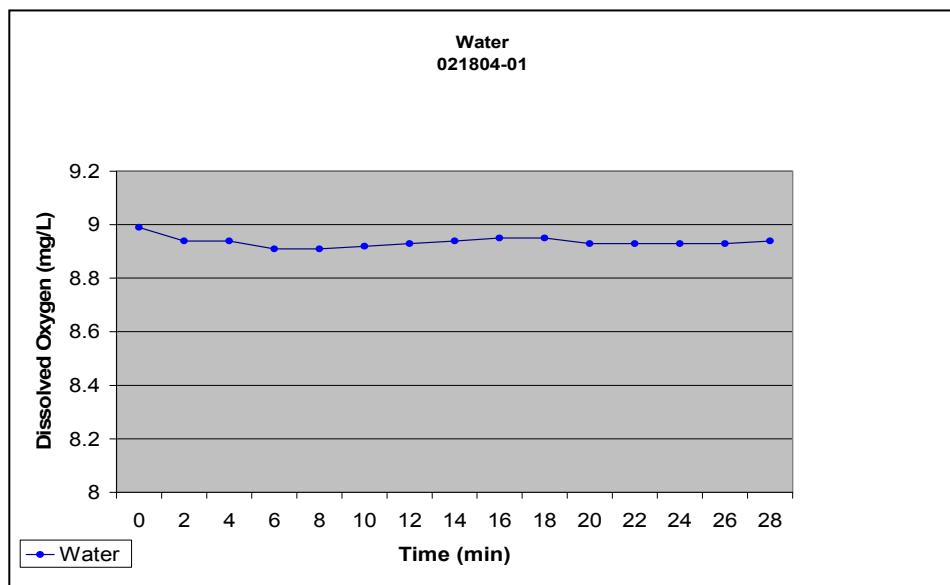
It was also observed that conventional DO probes in a closed system give a steadily decreasing reading for O<sub>2</sub>, evidently due to depletion of O<sub>2</sub> within the probe. A FOXY DO probe has been ordered that the manufacturer claims should not have this problem.

A FOXY-T1000 Fiber Optics Dissolved Oxygen Sensor System was received. This system uses the fluorescence quenching of oxygen molecules in our mud to measure dissolved oxygen. This DO probe is expected to have the important advantage over conventional probes in a closed system that it does not deplete O<sub>2</sub>.

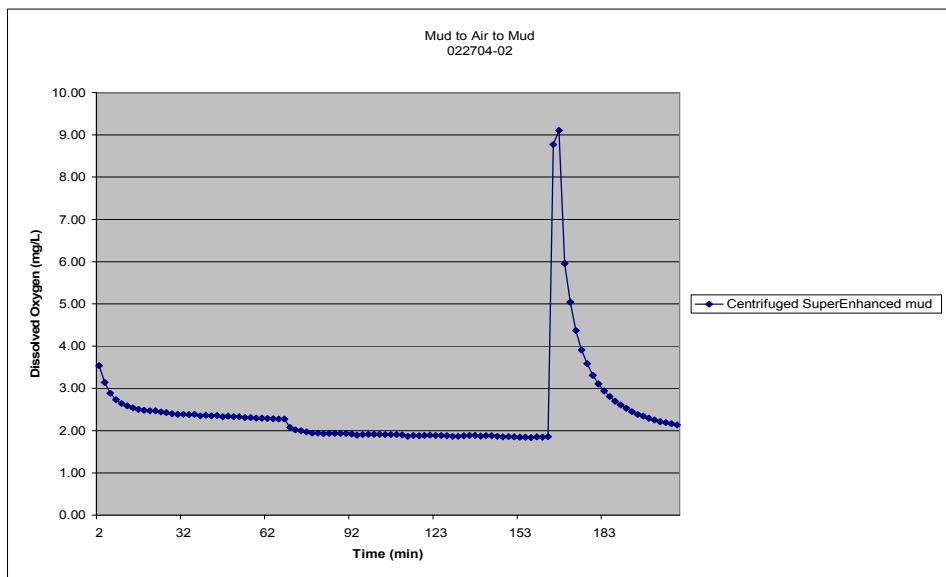
Initial tests were carried out to characterize operation of the new DO probe. Several tests were run to determine the accuracy of the probe and the effect of high-shear flow at the sensor tip; the latter is expected to reduce viscosity of the highly shear-thinning APHRON ICS drilling fluid and shorten the time to reach a steady-state value of DO.

Data on water and on air-free AphronICS mud (shown in the figures below) raised the following questions:

- ~Why are we obtaining such a low O<sub>2</sub> concentration (2 mg/L) in our mud, when theoretically we should be obtaining the same O<sub>2</sub> concentration as in water (9 mg/L)?
- ~With such a low concentration of O<sub>2</sub> being measured in our mud, might there be a reaction in the mud which scavenges or depletes the oxygen? Or is there a problem with the biocide in the mud which is allowing biological degradation to occur?
- ~What variables are affecting the response time of the FOXY probe?
- ~ Is viscosity playing a role in its measurement?



**Figure 11. Response of FOXY DO Probe in Fresh Water**



**Figure 12. Response of FOXY DO Probe in Fresh-Water Based SE APHRON ICS Mud**

One of the first attempts to understand why the DO concentration in APHRON ICS muds is so low entailed measurement of the Water Activity ( $A_w$ ) of the mud.  $A_w$  is a measure of the amount of unbound water, sometimes referred to as “active” water in the system. The results indicated that the APHRON ICS mud has the same water activity as that of pure water, therefore discarding the notion that the water in the mud is tied up in some way and is not able to accommodate as much dissolved  $O_2$ .

The FOXY probe is only sensitive to the environment at the probe tip. The sol-gel where the ruthenium complex is embedded is somewhat porous. The measured low values of DO suggest that mass transfer of  $O_2$  to the ruthenium complex is being impeded in some manner. We feel that there is a local phenomenon, such as formation of a low-permeability film, that may delay the probe’s response to the point that it is affecting the  $O_2$  equilibrium reaction at the sensor.

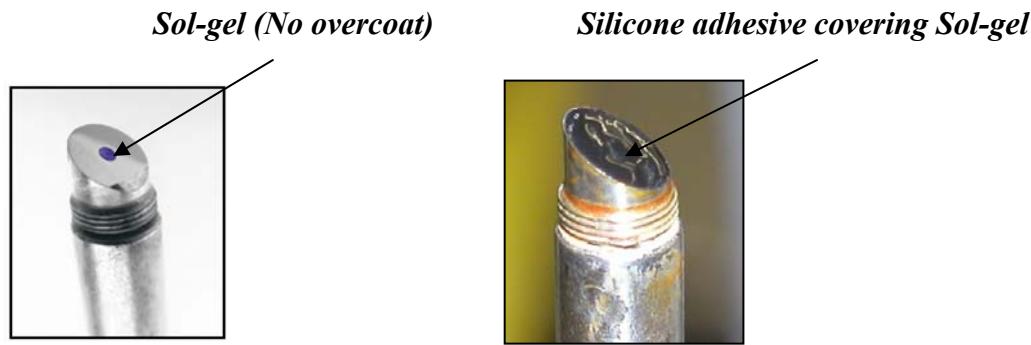
Data gathered with and without biocide indicates that there is no biodegradation phenomenon responsible for this effect. We are still investigating which component or combination of components may be creating this film affect.

The response time of the FOXY probe is limited by the speed of diffusion of oxygen into the sensor. In viscous samples, such as ours, the diffusion through the sample will determine the response time. Quite likely viscosity is playing an important role in this regard. Might it also be affecting the  $O_2$  equilibrium at the sensor? Data provided by the probe supplier indicates that high viscosity fluid, such as tree sap, does not affect the equilibrium reading of DO. Nevertheless, there may be some little understood viscosity-related phenomenon that could be responsible for the low  $O_2$  readings in the APHRON ICS mud.

There are no easy answers to the questions posed above. The FOXY probe does not appear to be failing when exposed to the APHRON ICS mud. When the probe is removed and re-exposed to room air or to water, it still gives the correct readings for both media. Something is affecting the probe immediately after it is immersed in the mud. A sample of the mud was sent to the manufacturer, which has confirmed these same findings.

#### *A Problem with the DO Probe*

Although the sensor itself appears to be functioning correctly, the silicone overcoat on the FOXY probe appears to be failing. At the high pH of our system (generally around 10), the silicone overcoat on the FOXY probe (which protects it from ambient light, chemical attack, and difference in refractive index of the environment) is being destroyed (see Figure 13). If the silicone overcoat is damaged, then the sol-gel complex used for the fluorescence quenching is slowly being destroyed as well.



**Figure 13. Effect of Aphron ICS Muds on Integrity of FOXY DO Probe**

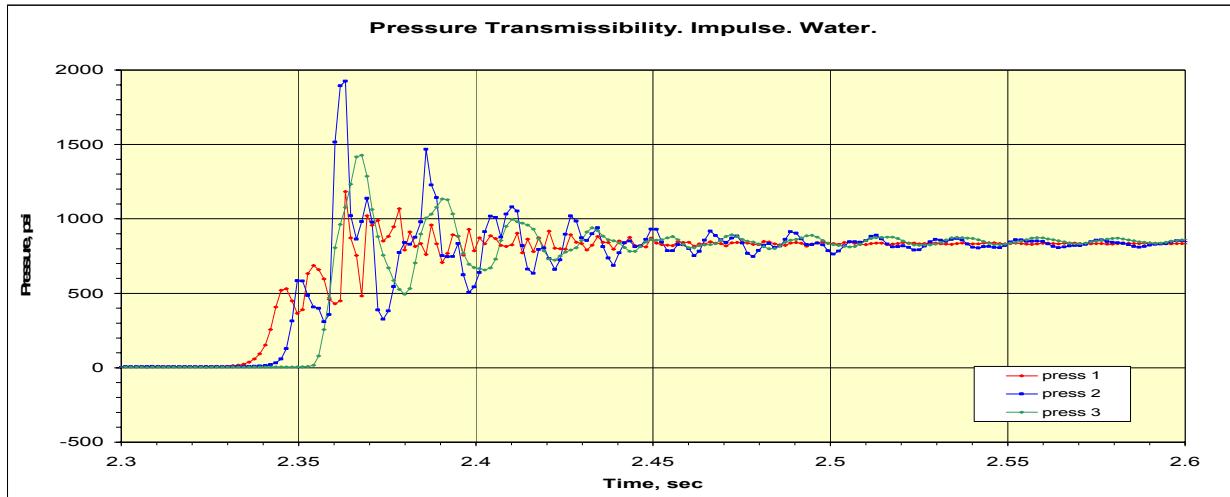
### Pressure Transmissibility

The old manually operated Ruska pump was replaced with a new ISCO D-500 syringe pump, which permits much more precise control of the pressure, speed of pressure change and flow rate.

Preliminary tests were conducted with a 6-m (20 ft) section of the system, using water. Three pressure transducers were mounted on the tube at 0, 10 and 20 ft from the pump. The test results indicate that the pressure change in the system occurs much faster than the time response of the pressure transducers. Propagation of acoustic waves and their reflection from the end of tubing produced complicated results which can not be used for pressure transmissibility determinations (see Figure 14).

A few tests were conducted in the 20-ft 0.635 cm ( $\frac{1}{4}$ ") OD system. The simple test protocol involved simply applying a pressure increase from ambient to 13.8 MPa (2000 psia), holding for several minutes, then depressurizing at the same rate. Two aphron drilling fluids containing 5%

and 38% air by volume were used. Both muds produced essentially the same results, indicating that the observed behaviors were the result of the base fluid and not related to the aphrons.



**Figure 14. Pressure Pulses through Fresh Water**

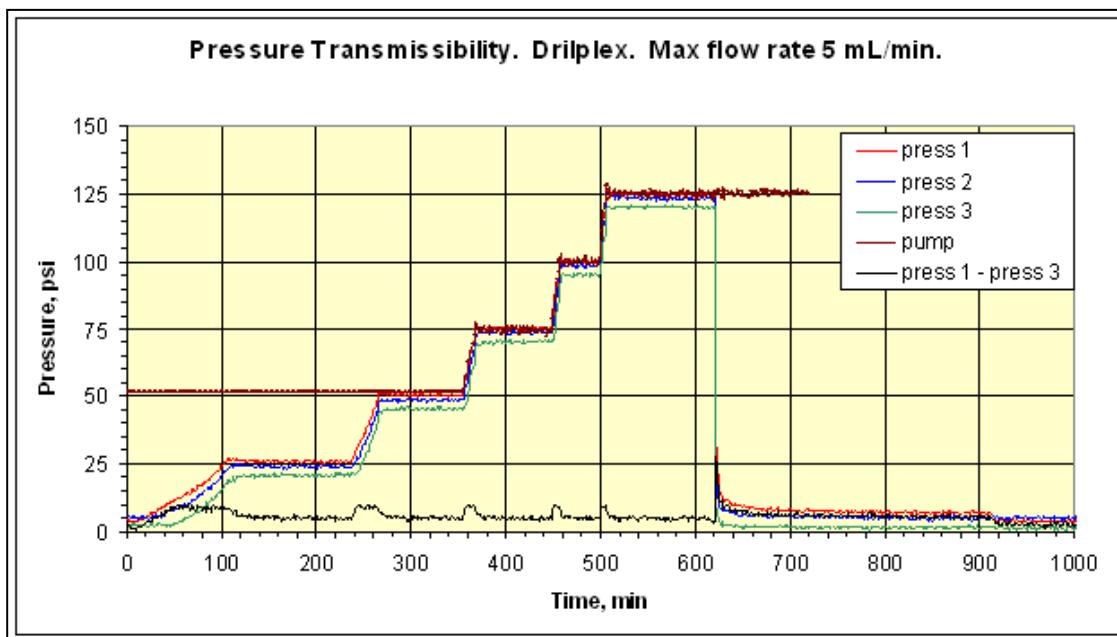
Some steady-state pressure drop tests were also carried out over the pressure range 0 to 13.8 MPa (2000 psia). Immediately after decompression, some small bubbles (aphron-size) appeared, but some big air bubbles also formed gradually and continuously. This indicates that some of the aphrons survived pressurization to 2000 psia. The large bubbles evidently consisted of air that had escaped from the aphrons during pressurization and dissolved in the base fluid. When the fluid was depressurized, the air simply came out of solution; the process was slow enough that the bubbles grew much like crystals growing from a supersaturated salt solution.

The test protocol for initial evaluation tests was changed from examination of high pressures to low pressures to ensure that the aphrons would be stable. The simple test protocol involved applying a pressure increase from ambient to 0.86 MPa (125 psia), stepwise in 0.17 MPa (25 psi) increments, hold for several minutes at each pressure, then depressurize quickly from 125 psia to ambient (0.101 MPa, or 14.7 psia). The pump flow rate was limited to 5 mL/min to ensure pressure equilibration during pressurization.

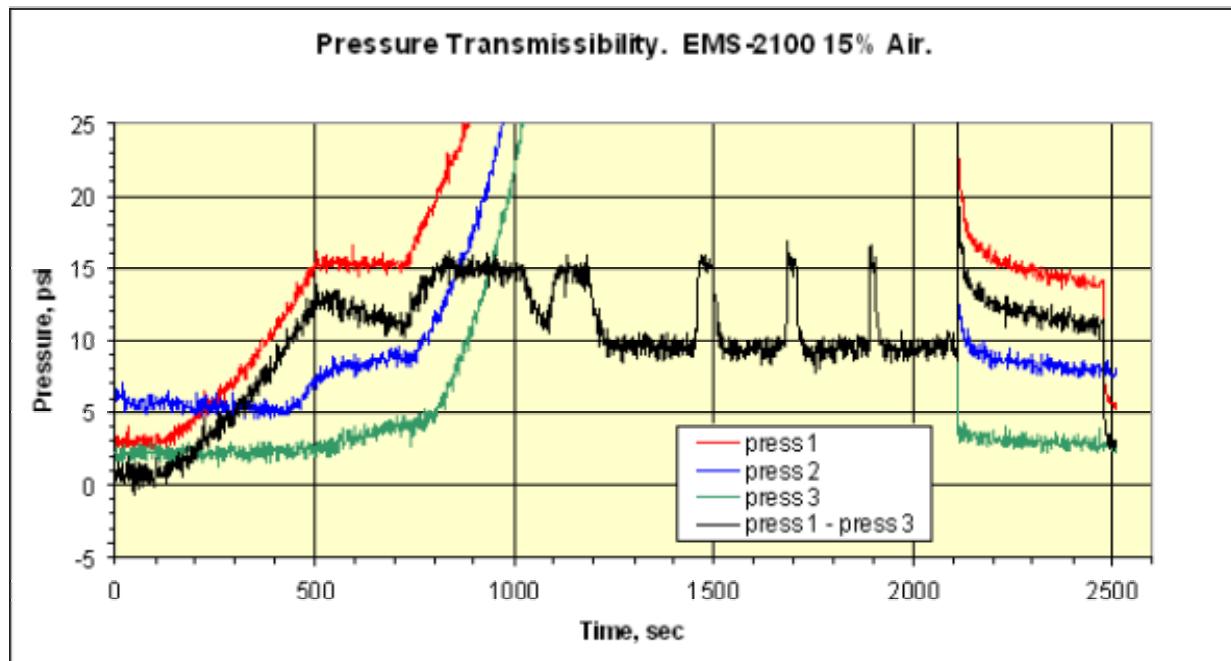
Two APHRON ICS drilling fluids (the standard polymer-based system) containing 5% and 38% air by volume and two conventional muds (DRILPLEX and FLOPRO NT) used for drilling in de-

pleted fields were used. The DRILPLEX test data are shown in Figure 15. Also, the new clay-based aphon drilling fluid, EMS-2100, was used for comparison (see Figure 16). Both APHRON ICS tests produced essentially the same results. The result for EMS-2100 was close to those obtained for the APHRON ICS fluids. The DRILPLEX mud showed a lower pressure drop than the APHRON ICS muds, while the FLOPRO NT mud behaved like water and did not produce any pressure drop in our experiment. The results, summarized in Figure 17, indicate that the APHRON ICS mud produces a greater pressure drop than the reference muds, but that the results are due to properties of the base fluid and are not related to the presence of aphrons. The pressure drops recorded between 0 and 20 ft from the pump (Press 1 – Press 3 in the figures below) were as follows:

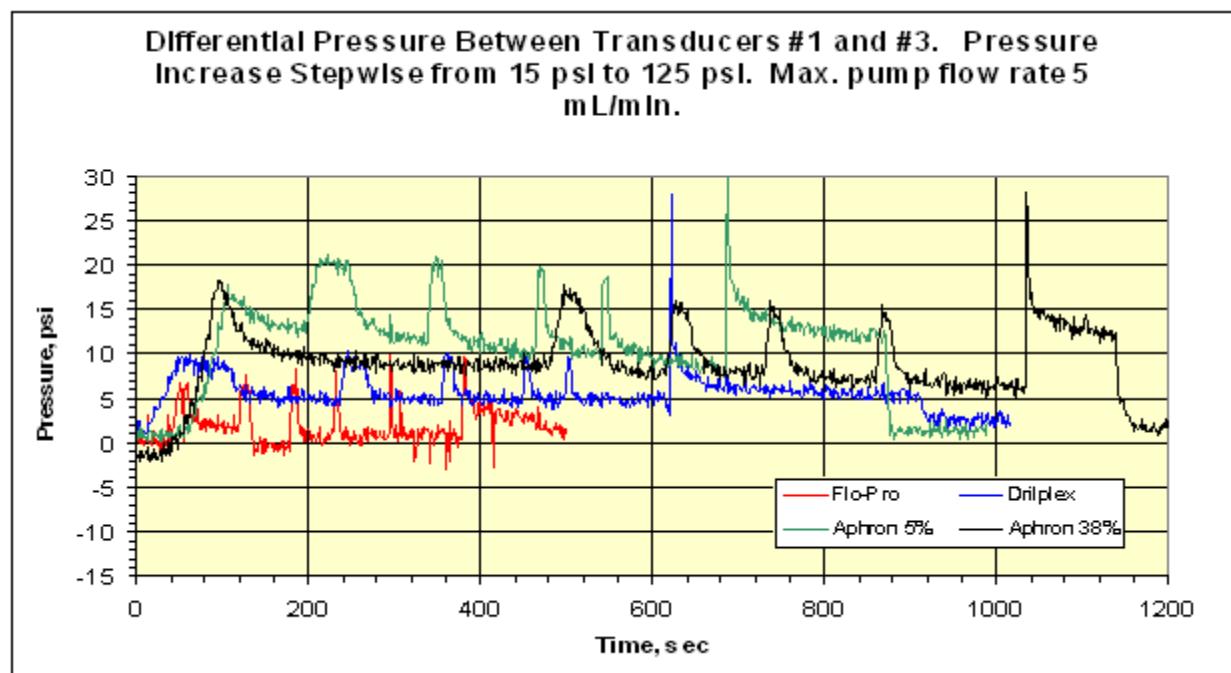
APHRON ICS (5% and 38% Air)	10 - 12 psi
EMS-2100	10 psi
DRILPLEX	5 – 6 psi
FLOPRO NT	1 – 2 psi



**Figure 15. Pressure Transmission through a DRILPLEX Mud**



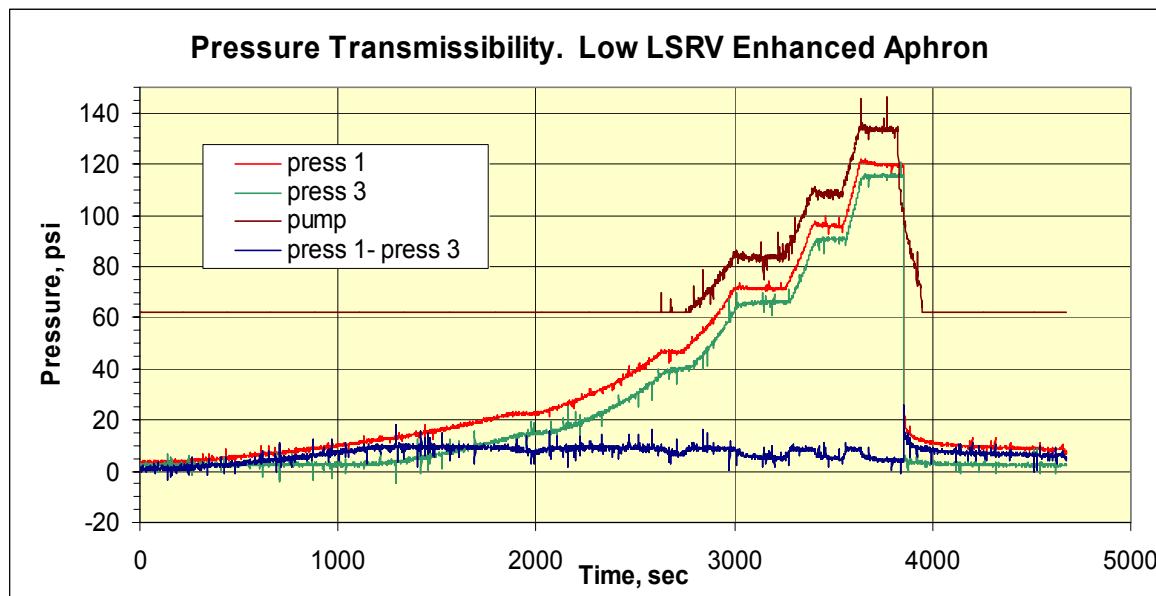
**Figure 16. Pressure Transmission through EMS-2100 (Clay-Based APHRON ICS) Mud**



**Figure 17. Pressure Drop Data for FLOPRO NT, DRILPLEX and Two APHRON ICS Muds**

To investigate the relationship between viscosity of the base fluid and pressure drop in our system under the conditions described above, an APHRON ICS mud with low LSRV was prepared. This sample, which contained 3 lb/bbl Go-Devil rather than the usual 5 lb/bbl, had an LSRV in the range of 104000 -118000 cP, which is about half the LSRV of the standard APHRON ICS fluid.

Pressure transmission experiments with this mud showed a pressure drop in the 6-m (20-ft) tube of 0.034 - 0.055 MPa (5 to 8 psi), which is, as expected, about half of the 10 – 12 psi pressure drop observed for the standard APHRON ICS mud.



**Figure 18. Pressure Transmission in a low-LSRV APHRON ICS Mud**

Unfortunately, the old data acquisition system began producing a very noisy background (see Figure 18), which made precise determination of the pressure drop between the sensors very difficult. New power supply and acquisition boards were acquired, along with a computer. The new system has been checked and is ready for use.

The hypothesis that is at the heart of this project assumes that aphrons accumulate in a fracture or pore throat to form a foam-like structure, which plugs the opening and distributes the stress uniformly. Several attempts were made to produce foam *ex situ* from the APHRON ICS mud, using variations in the mud composition – particularly the primary surfactant Blue Streak – and the air/mud mixing technique. The latter included various types of blenders, pumping air through the mud, and passing the mud and air through a small orifice and through fine screens. However, thus far the maximum air concentration obtained in the mud has been 60%, and this is clearly a dispersion of fine spherical bubbles rather than a true foam. An in-line homogenizer (a tube packed with coarse steel wool) will be tested next. Perhaps the most promising idea, though, is to use a long tapered tube through which a mud with a high concentration of aphrons is flowed; the aphrons are allowed to plug the outlet, thus forming the foam-like micro-environment *in situ*.

## CONCLUSIONS

The Aphron Visualization tests indicate that the Acoustic Bubble Spectrometer (ABS) provides measurements of Bubble Size Distribution (BSD) that are skewed to much lower values – as much as an order of magnitude – than the BSD obtained from Laser Light Scattering and Photomicrographic measurements. Nevertheless, it may be possible to incorporate an algorithm that serves to normalize the ABS data to provide a correct BSD. Also, the design of the HTHP ABS Cell recently constructed to carry out BSD measurements at elevated temperature and pressure must be changed to ensure safe and reliable operation.

For the Aphron Air Diffusivity tests, conventional membrane-capped DO probes have not proven suitable for measuring the kinetics of the transport of air out of aphrons at elevated pressures. A promising fluorescence probe appears to be experiencing similar difficulties. A galvanic technique typically used to monitor corrosion is being examined as a possible alternative.

The equipment being used to carry out the Fluid Density task is giving results that are too erratic to be quantifiable, and modifications are being made to increase its reliability. Nevertheless, some interesting results have come to light with respect to the survivability of aphrons under downhole conditions. Hysteresis in fluid density measurements during repeated pressurization/depressurization cycles suggests that some aphrons survive at pressures as high as 1000 psi, and the destruction and regeneration of aphrons are very time dependent phenomena.

The Pressure Transmissibility tests with aphron drilling fluids are showing clearly that, although aphrons may provide a small cushioning effect, most of pressure drop observed in long-path-length conduits (such as a fracture or high-permeability porous material) is caused by the high viscosity of the base fluid. It is also clear that aphron drilling fluids provide a greater pressure drop than other low-invasion drilling fluids, such as FLOPRO NT and DRILPLEX. The current focus of this task is on pressure transmission through an aphron network, such as that expected to be created at a fracture tip or pore throat during invasion of the drilling fluid into a rock formation.

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## LIST OF ACRONYMS AND ABBREVIATIONS

ABS = Acoustic Bubble Spectroscopy

APHRON ICS = Aphron Invasion Control System

Blue Streak = Surfactant package which serves as aphron generator for the APHRON ICS system

BSD = Bubble Size Distribution

DO = Dissolved Oxygen

EMI- = Experimental M-I *LLC* product

FloVis Plus = Xanthan Gum polymer

HTHP = High Temperature and High Pressure

OD = Outer Diameter

PVC = polyvinyl chloride