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Injection and Tracking of Micro-Seismic Emitters to Optimize Unconventional Oil and Gas (UOG) Development

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DISCLAIMER:

ABSTRACT

The goals and objectives with this project are to develop fracture and fracture proppant mapping and monitoring technologies that will facilitate both efficient and environmentally prudent development of Unconventional Oil and Gas (UOG) resources. We will achieve these concurrent objectives by developing two novel technologies and then deploy these technologies in an Unconventional Oil and Gas (UOG) well before, during and after a formation fracturing operation. The first of these technologies is Injectable Acoustic Micro Emitters (IAME or AME) that can be mixed with proppant in small or very small, equal to or less than 1%, concentrations by mass and volume, and injected into newly created hydro fractures concurrent with the proppant. The second enabling technology is an ultra-sensitive large-bandwidth large-aperture Fiber Optic Seismic Vector Sensor (FSOVS) array that can be deployed in both vertical and horizontal wells. The IAME/AME mixed with the regular proppant will generate small, pressure actuated, seismic signals at a delayed time after the proppant injection has stopped and after the proppant – IAME/AME mixture has reached deep into the fracture network. These acoustic signals will allow tracking the actual location of the proppant and compare these locations with the location of the fracturing of the rock. This data will also allow tracking the width and radial extent of the fractures by mapping the number of acoustic signals from each 3D location.

The Fiber Optic Seismic Vector Sensor (FOSVS) array will also record the sounds from the casing perforations that are created at known locations. The seismic data from these sources at the known locations will then be used to calibrate both the seismic acquisition system including the vector orientation of the 3C pods as well as generating a 3D velocity model. The FOSVS system will also monitor the hydraulic fracturing process and the injection of the proppant into the fractures during the reservoir stimulation. It will do so by recording and locating first the micro seismic data generated during the fracturing of the rock making up the oil and gas reservoir formation and, second, the flow of the proppant and associated fluids and third by recording and locating the time-delayed micro seismic data generated by the Injectable Acoustic Micro Emitters (IAME/AME) that were mixed with the proppant prior to injecting the mixture into the hydro-fractures.

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REPORT DETAILS

We have considered and tested Injectable Acoustic Micro Emitters (IAME/AME) based on two different technologies developed by two companies. The two companies are Fluidion SAS located in Paris, France and Terves, LLC located in Euclid, Ohio, USA. The smart acoustic proppant particles from either of the two companies will be mixed with regular proppant particles at heavy dilutions. A typical single hydro fracturing stage in the Marcellus shale uses about 250,000 lbs. (125 short tons) of proppant. The dilution depends on which Injectable Acoustic Micro Emitter that is used. Fluidion estimate that 1,000 to 2,000 of their Acoustic Micro Emitters (AME), might be sufficient for one hydro fracturing stage. The size of the Terves IAME is considerably smaller. The size of the Terves IAME/AME matches the size of the proppant now preferred by the UOG operators which is reported to be 100 mesh or 150μm in diameter. The diameter of the IAME/AME is 150μm, so a 1% mix by weight, for a total of 2,500 lbs. or about a metric ton, of the Terves IAME would represent many billions of IAME particles. The energy from each individual Terves IAME is smaller than the Fluidion AME but the energy will increase by superposition of the acoustic events.

The Terves Injectable Acoustic Micro Emitters (IAME's) have the same size as the proppant, typically either 40-70 or 100 mesh and are likely to be the better option just from a size point of view. The IAMEs from Terves will be mixed with the regular proppant at a concentration of no more than 1% of the total injected proppant weight in a fracture. If we inject 250,000 lbs. of proppant we will thus mix the proppant with 2,500 lbs., or less, of IAMEs from Terves. The smart Terves IAMEs have no role in propping the fracture, which will be done using the regular proppant, but need to be capable to withstand the injection process and the associated pressures and stresses appearing in the proppant pack. Furthermore, the IAME must be small enough to be effectively be injected with the proppant. The size of the Terves IME's is between 150 - 420 μm while the size of the Fluidion AME's range from 2 – 4 mm. The IAME's from Terves are thus considerably smaller than the Acoustic Micro Emitters (AME) from Fluidion. If the AME's are too big they will not enter the fracture network and this greatly favors the Terves IAMEs'. The drawback with the very small Terves IAME devices is however two-fold. They generate only a small amount of acoustic energy, a 10 or so micro Joules, and the energy generated is very high frequency. We might be able to overcome this using sophisticated signal processing technologies.

The Fluidion and Terves Acoustic Micro Emitters work in principle in the same way. The Injectable Acoustic Micro Emitters (IAME/AME) generate an acoustic signal some time after the proppant injection has stopped by collapsing a small cavity. The time delay of the cavity collapse must be tailored to the application during the manufacturing process of the emitters. A time delay of several hours has been achieved in our laboratory using the Terves Injectable Acoustic Micro Emitters (IAME). We have not tested the collapse delay function from Fluidion.

The delay function allows the proppant to be pumped into the formation, and the IAME/AME signal to be generated and recorded at a later time, when the pumping noise has dissipated. The acoustic signature of the proppant can be a simple signal, or a series of several signals in the case of multiple cavity proppant particles, which create known waveforms. This allows for improved signal recovery, leading to a better detectability.

To monitor the data from the Injectable Acoustic Micro Emitters (IAMEs) we have built a drill pipe deployed fiber optic ultra-large ultra-dense spatial sampling borehole seismic vector sensor array using technology invented and developed by Paulsson, Inc. on previous DOE funding. The borehole seismic system built under this program was projected to have up to 100 3C sensor pods spaced 25 ft. apart making the array section 2,500 ft. long. The array section will be attached to an existing 15,000 ft. fiber cable allowing the array to be deployed in a vertical or horizontal well to a drilled depth of about 17,000 ft. The array can operate at temperatures over 500ºF and at pressures up to 30,000 psi.

The borehole seismic system was built using Titanium and Inconel steels allowing the system to operate in corrosive and high temperature well environments. The ultra-large all-optical borehole seismic arrays will have no electronics in the borehole nor require electric signals or power making it the seismic array ultra-robust and intrinsically safe.

General Status

The Acoustic Micro Emitters (AMEs) from Fluidion were part of the original proposal. All joint FOSVS – AME work were using the Fluidion AMEs from December 2014 to 2017. In

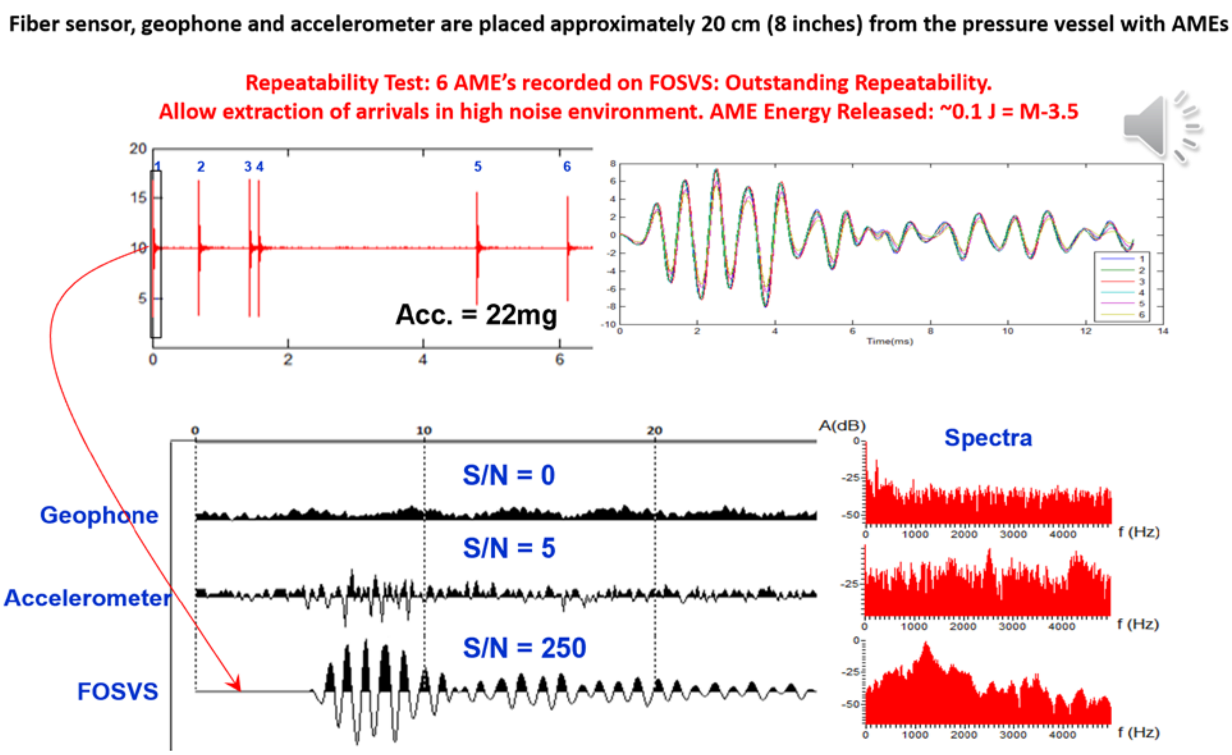

Figure 1. Test of Fiber Optic Seismic Vector Sensors (FOSVS), Geophones and Accelerometers using and Fluidion Acoustic Micro Emitters (AME)

December 2014, the Recipient successfully tested Fluidion's Acoustic Micro Emitters (AME's) together with the Fiber Optic Seismic Vector Sensors (FOSVS), see Figure 1, in a small-scale laboratory experiment and found that we can record data with a S/N ratio of 250 using our combination of sources and receivers. In the same time, as excellent data was recorded on the Fiber Optic Seismic Vector Sensors (FOSVS) a state-of-the-art geophone failed to record any data at all. The data recorded on the fiber optic sensors was extremely repeatable showing that both the AME source and the receivers generate and record repeatable data. The fact that we have a repeatable system will allow for both stacking as well as using correlation to detect arrivals in a high noise environment.

In September 2016, we successfully tested the Fluidion Acoustic Micro Emitters (AME's) together with the fiber optic sensors in a 30 ft long 15 ft wide pool and found that we can record excellent data with a S/N ratio exceeding 50 over a distance of 20 ft. This data is shown in Figure 2. To test a longer-range detection of the AME's by the FOSVS sensors, we

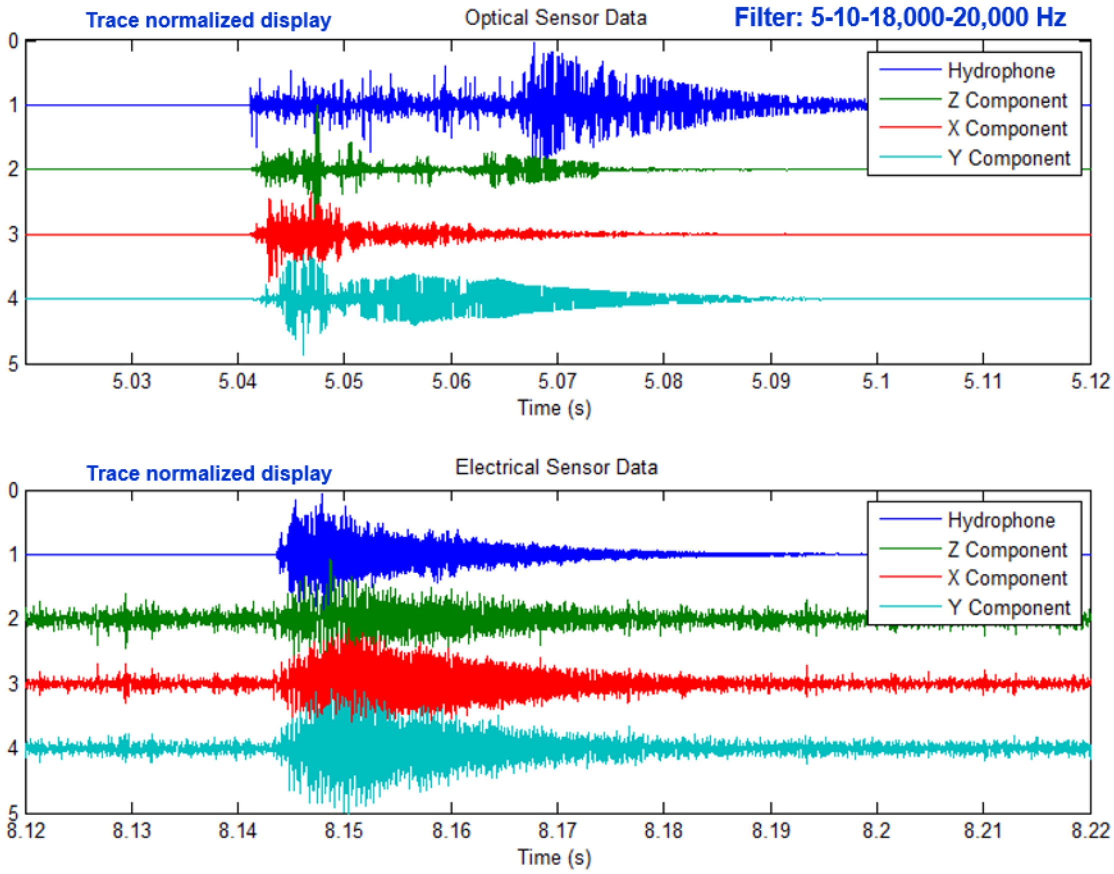

Figure 2. Fluidion AME tested at 4,000 psi in a pool with a transmission distance between sources and receivers of 20 ft.

conducted a lake test to record and characterize the AME generated data over larger distances. This test was performed in April 2017 in the 2017Q3 reporting period. We successfully recorded arrivals at distances over 100 ft despite very noisy conditions in the lake. We expect, given a quiet environment, that we will be able to record good arrivals at a distance of 500 ft or more using the AME as the acoustic sources.

In December 2016 we also successfully tested the Injectable Acoustic Micro Emitters (IAMEs) from Terves LLC without the delay function both in laboratory and pool environments

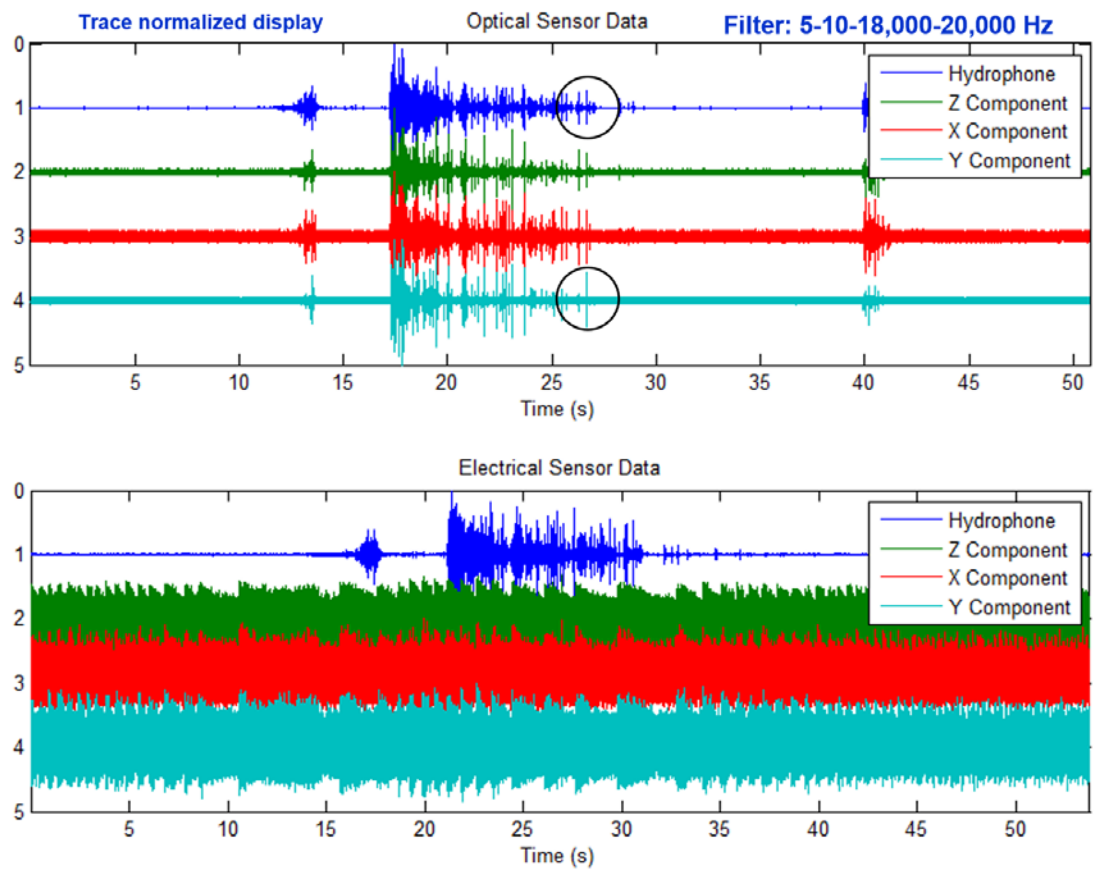

Figure 3. Test of 4 gram of Terves Micro Spheres rated at 4,000 psi. The arrivals can clearly be seen on the FOSVS sensors but not at all on the Geophones.

generating very strong and easily detectable signals. This data is shown in Figure 3. In 2019 we successfully tested the delay function of the Terves IAMEs. More work needs to be done to properly calibrate the time delay as function of reservoir temperature, reservoir chemistry and reservoir pressure but we have in principle demonstrated for the first time the delay function of IAME.

In 2019 we did a calibrated test of the Terves' AIMEs and found that the Terves' AIMEs generated energy in the range of 1 – 10 µJ. Properly coupled and in a low noise, high seismic Q environment we should be able to detect these events at a distance of more than 500 ft in the field at the reservoir level.

During this test we evaluated a total of six different collapsible hollow spheres as possible candidates for Injectable Acoustic Micro Emitters (IAMEs). We found that the Terves IAME's were equal or superior to all other choices. Due to their small size, about 150 – 250 µm, relative ease of adoption to different environments and the low cost the Terves IAME are likely the most viable alternative for a commercial Injectable Acoustic Micro Emitter (IAME) product. Given that Terves has developed a functional delay mechanism for their IAMEs we will continue to work with Terves on this promising proppant injection mapping technology.

Status of Fluidion's AME's (statement by Fluidion)

Hydrodynamic simulation of the timing circuit was performed under representative pressure and temperature conditions (Poiseuille flow simulation in rectangular channel geometry). Three timing circuit designs were cleanroom-manufactured out of silicon and glass, corresponding to 5min, 30min and 1hr timing delays.

Multiple AME device designs were realized (4), corresponding to different geometries (rod, cylinder, rectangular) and glass-silicon manufacturing process. FEM stress analysis was performed for expected hydrostatic and localized stress conditions. All-silicon manufacturing was performed as well, using a recently-developed eutectic bonding process for sealing the devices. This manufacturing technique offers better machining capabilities for complex MEMS and microfluidic devices, since the etching of silicon is much better controlled than that of glass. We also developed a new brazing process for sealing the devices that requires the use of a pick- and-place machine. This type of manufacturing allows tremendous amount of customization, since the devices are built on a one-by-one basis (automation possible) and therefore are not constrained by the batch manufacturing process requirements. Using the above techniques, two generations of high-pressure AMEs were manufactured in the cleanroom, ensuring the compatibility of the new bonding process with the ensemble of fabrication steps required for AME integration.

Fluidion designed and fabricated a proppant flow fixture from PMMA using laser cutting as well as 3D laser engraving. An actual fracture geometry was used (Hurst exponent: 0.8) and could be imprinted onto the fracture walls using laser ablation technology to simulate a real environment. A literature review was performed of all proppant transport and fracture geometry literature to date, and previous experiments were analyzed, and conditions adapted to the current setup. Transparent glass microspheres were used to allow the visualization of proppant, in conjunction with both opaque and photo luminescent mockup AMEs. We used widths from 1 to 12 mm, which are representative of actual fracture geometries recorded in the field. We also included tapered joints to allow for variable width fractures (6mm to 2mm and 3mm to 1mm). Different geometries of AME (cylindrical vs. rectangular, different sizes) were attempted, and the time-dependent distribution of proppant and AMEs was recorded using camera photos and movies. Test were performed in both smooth and rough geometries with different AME geometries (cylindrical, rectangular, rod). The experiments using this setup are ongoing. Significant amounts of data have already been collected and are currently being processed.

All-silicon manufacturing was performed as well, using a recently-developed eutectic bonding process for sealing the devices. This manufacturing technique offers better machining capabilities for complex MEMS and microfluidic devices, since the etching of silicon is much better controlled than that of glass. We also developed a new brazing process for sealing the devices that requires the use of a pick-and-place machine. This type of manufacturing allows tremendous amount of customization, since the devices are built on a one-by-one basis (automation possible) and therefore are not constrained by the batch manufacturing process requirements. Using the above techniques, two generations of high-pressure AMEs were manufactured in the cleanroom, ensuring the compatibility of the new bonding process with the ensemble of fabrication steps required for AME integration.

Two additional manufacturing runs were made, which involved full silicon devices using with large internal volume cavities, and devices incorporating a fluidic clock, for initial testing. Currently, tests with these devices are in progress, in order to qualify the clock functionality and the capability of the actuator to collapse the device at the end of the timing period. Tests were also performed at Paulsson, Inc.'s facilities, in conjunction with the FOSVS tool. A new generation of devices was manufactured for this purpose, and testing was performed in a large water tank (swimming pool or equivalent). This allowed the tool and devices to be fully decoupled, thus allowing representative data to be obtained in laboratory-controlled conditions, as a pre-field experiment. For a more detailed description, see description of Task 12.0 results.

Fluidion Acoustic emission estimate for smart proppant particles (AME)

Energy release by rupturing cavities: The total energy E emitted during the rupturing of a solid particle of lateral size l containing a hollow internal cavity of volume $V = l^3$ and subjected to a fluid of pressure p is equal to the total work performed by the fluid during the collapse, $E = pV$. This energy is released mainly in two forms: acoustic energy (E_{ac}) and heat (Q). The large majority of the energy is released as acoustic energy, since the collapse process is extremely fast and thus adiabatic. We will therefore ignore heat losses and assume the following approximation: $E_{ac}=pV$. For a typical smart proppant of size l=1 mm and volume V=1 mm^3 rupturing at a pressure of 10,000 psi, this corresponds to a total energy release E_{ac} ~0.1 J, the equivalent energy of a Moment Magnitude M-3.5 event on the Moment Magnitude scale. This can be increased to a Moment Magnitude M-3 event if the lateral size of the rupturing cavity is increased to l =1.8 mm. Since the total energy released is also dependent on the hydrostatic pressure in the fracture, operational conditions will affect the effective event magnitude.

Paulsson Fiber Optic Seismic Vector Sensor (FOSVS) System Status

We have successfully designed, prototyped and laboratory tested the fiber optic seismic vector sensors at different frequencies and at different temperatures. We have compared the fiber optic

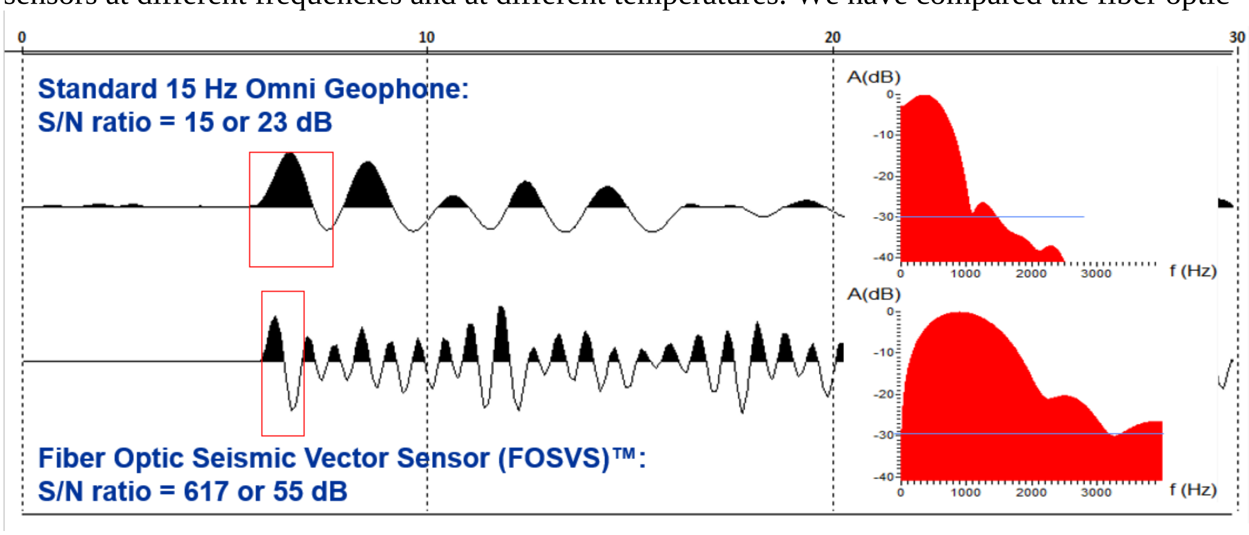

Figure 4. Fiber Optic Seismic Vector Sensor (FOSVS)™ vs. 15 Hz Geophone. Data recorded simultaneously from a single tap test. Sampling rate: 8,000 Hz. High cut filter at 2,500 Hz. FOSVS S/N ratio is 41 times higher than S/N for Geophone and FOSVS -30 dB point is 3,300 Hz vs 1,100 Hz for Geophone

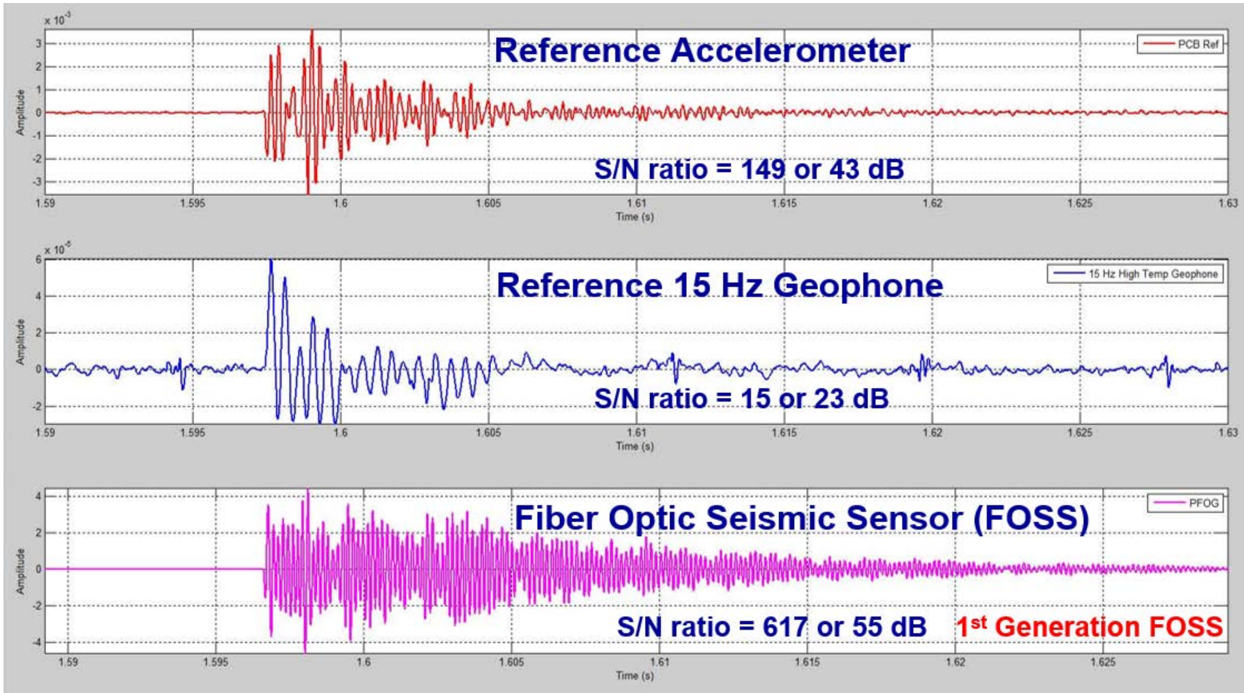

Figure 5. Seismic Traces from Tap Test of Three Sensors. Band Pass Filter: 5 – 2,500 Hz

seismic sensors with state-of-the-art accelerometers and state of the art geophones. In each case our fiber optic seismic sensors record significantly better data than the legacy geophone sensors. See Figure 4 and 5 that shows the result from comparative testing.

We have specified, selected and purchased the sensor fiber. We have specified, selected and purchased the Fiber Bragg Gratings (FBG's) for the sensors. We have also developed a manufacturing capability internally to Paulsson for the manufacturing of our specialized FBG's.

We have completed the manufacturing of 300 Fiber Optic Seismic Vector Sensors (FOSVS) that will be used in the 100 level 3C array. The sensor manufacturing, and assembly processes are still being perfected as of this writing to improve the sensitivity of the sensors. This perfection process will continue long after this project has been concluded.

We have successfully tested and tuned the interferometric interrogator that will be used for the fiber optic seismic system. We have upgraded the interrogator with a new set of Erbium Doped Fiber Amplifiers (EDFA's). We have completed the design of the interrogator and we are in the process of manufacturing a prototype unit that can operate 300 seismic channels using fiber optic sensors. This is the scope of a separate project.

We are investigating a new data telemetry approach to further lower the noise in the data. We have completed the manufacturing of the 110 sensor pods providing 10 spare units for the 100-level array.

We are continuing the manufacturing of the sensor pod housings. We have thus far completed 40 units. Significant effort was expended on the programming of the CNC machines that are producing the sensor pod housings.

We have upgraded our field processing capabilities, so we are now able to record and process both micro seismic and VSP data in real time.

We have completed the design and construction of a sound insulated temperature-controlled instrument room in our field deployable spool container to reduce the field environmental noise for our optical instrumentation. This effort was completed in June 2017.

A field comparison in a shallow borehole in August 2017 of the data recorded with the FOSVS sensors and standard 15 Hz geophones showed that the FOSVS sensors are much more sensitive and record broader band data from the same source.

Paulsson-Battelle Project Summary

Recent, August 2019, processing of the micro seismic data from a survey with Battelle in 2016 recorded in a Michigan reef shows an extremely promising correlation of injected pressurized fluids and micro seismic data recorded by the FOSVS sensors. Battelle Memorial Institute contracted with Paulsson, Inc. in 2016 to monitor and characterize over a period of one month the injection of CO2 into the Dover 33 reef located in Michigan operated by Core Energy LLC. The data recorded at the Dover 33 reef show a clear correlation between Pressure (P) and Micro-Seismic (MS). In particular there are three rapid pressure increases that each generate an increase in the MS events: As seen in Fig. 6, the dates for the three Pressure/Micro Seismic events are 6/20, 6/30-7/1, and 7/7-7/8 all 2016.

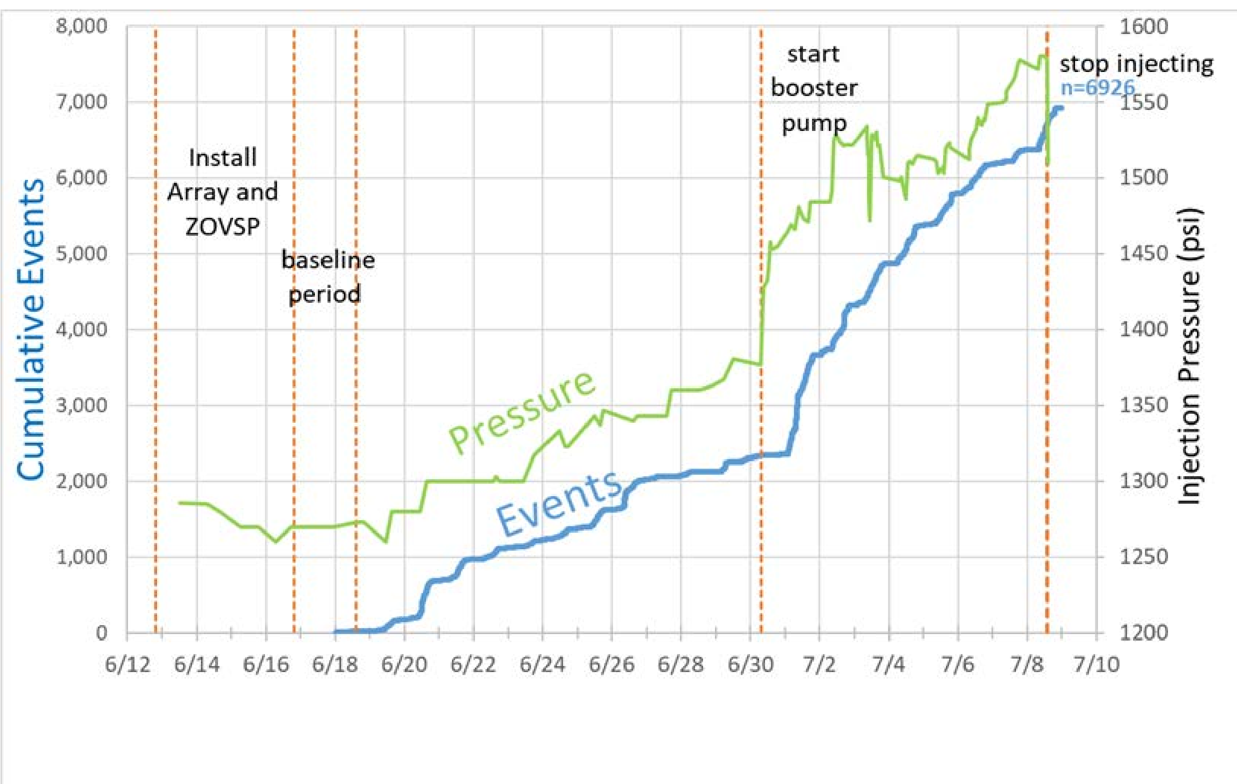

Figure 6. Cumulative Micro Seismic Events and CO2 Injection Pressure as function of time at the Dover 33 Reef.

Paulsson's ultra-sensitive broad-band high-temperature and robust Fiber Optic Seismic Vector Sensor (FOSVS) were developed under the DE-FE0024360 project to use in UOG operations. Other applications include CCUS and EGS operations, wastewater injection, and the monitoring of earthquake faults and Gas Storage Fields.

WHAT WAS ACCOMPLISHED

Budget Period I

Task 2.0: Preparation and Initial Design of the AME's

Paulsson will analyze the environmental conditions of pressure and temperature corresponding to hydraulic fracturing jobs in the geographical area of the future field test. The types and sizes of proppants being predominantly used will be determined by the Recipient, as well as the characteristics of the hydraulic fluids including but not limited to viscosity, density, yield stress, surfactant, and additive concentrations. The Recipient will base the analysis on literature review and information provided by oil industry partners to the project field study partner. The Fluidion completed analysis will enable drafting the principal (AME prototype) specifications of the testing fixtures to be designed and resulting AME's. This information will be included in a report titled the AME's prototype specification report and provided to the TPO per the details provide in the deliverable section of this SOPO.

Fluidion will design the high pressure, high temperature (HPHT) microscopy bench to be capable of performing direct microscopic observation under pressure up to and including 10 kpsi and temperature up to and including 212°F. These temperature and pressure conditions are to be representatives of the fracturing fluids that will be used during field testing and at a minimum be similar in viscosity, density, yield stress, surfactant and other additives' concentrations as defined by the specifications listed in the AME's prototype specification report.

HPHT test bench fabrication will be performed by Fluidion using high-strength materials that are compatible with the fluids being used during testing such as, but not limited to, high strength alloy body and cap capable of operating in brine, Hydrogen chloride (HCl) or Carbon Dioxide (CO_2) environments, and sapphire optical window for microscopy observations during the bench test, designed to operate safely (minimum safety factor of 1.5) at pressure up to and including 10 kpsi and temperature up to and including 212°F.

Fluidion will design and fabricate a proppant flow fixture from transparent plastic, such as but not limited to Poly methyl methacrylate (PMMA) or equivalent, to allow the visualization of proppant and AME injection and distribution within simulated planar fractures with lateral dimensions ranging from 1 to 10 mm and extending between 0.25 and 1.0 meters. Fluidion designed, and fabricated flow fixture will also include tapered fracture channels designed within the defined dimensional specifications.

Fluidion will optimize the AME size and shape to ensure homogenous transport within the fracture and a representative spatial distribution. Fluidion will complete a study of propagation distance vs. AME shape and size, and fracture width. Fluidion will manufacture the flow fixture that meets or exceeds safe operating conditions using transparent plastic materials such as but not limited to PMMA or equivalent, that provides relatively high strength and excellent clarity.

Fluidion will develop a minimum of four and a maximum of six AME device designs.

Fluidion will use finite element analysis simulations tools to model the stresses in the device under a minimum of three types of mechanical constraints. These constraints are listed as follows but are not limited to;

1. Isotropic loading resulting from increased hydrostatic pressure representative of device deployment in pressurized fluid;
2. Localized stress due to hydraulic actuator action which is responsible for ultimate device rupture and acoustic emission;
3. Uniaxial stress resulting from potential fracture-closure stresses.

Fluidion will complete hydrodynamic simulations for timing circuit operations for different fluid rheologies that are characteristic of the fracturing fluids expected to be used, as well as simulations of the cavity fill-up after rupture. These different fluid rheologies and characteristics will correspond to the specifications drafted in the Specifications Report, and as a minimum will include fluids with a minimum of two different viscosities, and fluids with normal and shear-thinning behavior. Fluidion will generate a statistical distribution of AME trigger delays that respond best to the operational parameters of the hydraulic fracturing process at a minimum for total fracturing duration per stage and time between stages. Fluidion will generate complete design and fabrication process specifications for manufacturing a minimum of four different shapes and a minimum of two different sizes of AME's. These are defined but are not limited to the following:

- Cubic shape, 20/40 mesh size AME
- Cylindrical shape, 20/40 mesh size AME
- Cubic shape, 40/70 mesh size AME
- Cylindrical shape, 40/70 mesh size AME

At a minimum, for the four general categories listed above, Fluidion will develop design variations for the purpose of, but not limited to, optimizing the resulting acoustic signal and for understanding the dependence of acoustic signatures on geometric parameters, such as but not limited to, cavity shapes.

Rod-type AME's with multiple cavities (between 2 and 4) will be designed by Fluidion, as direct extensions of the above minimum four designs.

Fluidion will perform initial cleanroom process qualification and development to fabricate the AME designs developed above and will initiate cleanroom fabrication of first prototypes and test runs corresponding to one of the above minimum four designs.

In 2016 Paulsson started to investigate the Terves Injectable Micro Emitters (IME's) as an alternative to the Fluidion technology. The small size and the low-cost favors using Terves technology for our field trial. One concern is the small energy and high frequency signals generated by the Terves IME's.

Achievement:

Fluidion performed literature review regarding fracturing fluid composition and additives and have concluded that the current trend in shale fracturing is the use of slick water, with viscosities similar to that of normal water (~1cP). This information was confirmed by information acquired during field visit in Aug. 2015 to the Fayetteville shale field (Southwestern Energy). We obtained samples of actual sand and resin coated sand proppant particles from field locations in TX, as well as 40-70 and 100-mesh proppant from the SWN sand plant (actual proppant used in all Fayetteville locations). The additional field constraints were assessed. Ideal AME injection location was identified at the blender site. From blender to well the AMEs need to survive pumping through the centrifugal discharge pump and the high-pressure positive displacement pump. Initial experiments using centrifugal pump in Fluidion's facility showed excellent survivability of the AMEs.

Fluidion performed a flow loop test using the actual field equipment (positive displacement pump truck) in December 2016 at the SWN Fayetteville equipment base. The SWN completions manager in charge was Grant Jacobs, and the equipment and field location crew were managed by Ted Hunter. The field personnel involved: John Grant, Nathan Hales (pump) and Justin Bolding (blender). Dan Angelescu from Fluidion also participated. The flow loop consisted of pumping water from a storage tank to the blender, where it was mixed with Fluidion mock-up AMEs and injected in the high-pressure pumping trucks. From here the water was discharged back to the storage tank through a 100-micron sock filter, which retained all fragments of the actual AME's at the exit of the high-pressure pumps.

Different types of mockup AMEs were manufactured, of cubic shape and with two characteristic dimensions (app. 2mm and 3mm nominal) in two types of material (hard silicon and glass, and softer plastic respectively - PMMA). These were manufactured in the cleanroom (hard devices) or, respectively, using a laser cutting process for the PMMA devices. Exact dimensions used:

- Silicon-glass device 1.8X1.8X1.6mm^3
- Silicon-glass device 2.9X2.9X2.6mm^3
- PMMA 2X2X1.8mm^3
- PMMA 2.6X2.6X2.7mm^3

The test procedure involved starting the pumps (centrifugal pump at blender and high-pressure positive displacement truck pump), waiting for the flow to stabilize at 100bbl/min, and injecting different batches of the AME devices progressively (quantities injected were recorded). After a few minutes, the pumping was stopped, and the eluted devices were recovered from the filters. These were counted and stored for further analysis.

The first conclusion of this test was very positive: a sizeable fraction, about 25% currently, of the Fluidion AME devices in the size range investigated can survive the pumping procedure that would inject them into the formation. It is important, however, to inject 3X-5X the number of devices that are expected to emit an acoustic signature downhole, since some of the silicon devices will be destroyed (partially or completely) by mechanical stresses from the positive displacement pumps. A second conclusion is that a larger percentage of PMMA devices survive the pumping, which is accounted by the ductile nature of the plastic material: PMMA can accommodate stresses without the brittle breaking that is characteristic of the harder materials.

Fluidion designed and manufactured a high-pressure high pressure, high temperature (HPHT) microscopy bench rated and pressure tested to 10,000 psi, capable of performing direct microscopic observation using their existing LEICA inverted metallurgy microscopy setup. The bench used high strength steel for the housing, a sapphire window sealed with Viton O-ring for the optical port, and bronze cap. All the materials are qualified for continuous operation at pressure at temperatures to 170°C, and will withstand occasional operation to 200°C. These temperature and pressure conditions are beyond what is typically met in hydraulic fracturing wells.

A new high-volume pressure testing chamber was designed and built by Fluidion, with a total volume of 20L (5.3 Gal) and 10,000psi capability. The upper cap of this pressure chamber was outfitted with electrical feed-throughs and the capability to later-on incorporate optical fiber feed-throughs. In addition, four sapphire windows were provided, which allows real-time speed camera visualization of the high-pressure testing. The pressure chamber was installed in the Fluidion facility on a custom-made chassis, and it was powered with a pneumatic-drive hydraulic high-pressure pump. This chamber will be used for representative acoustic experiments using large proppant populations, to qualify devices for further testing.

An initial batch of test AME was designed, manufactured, and laboratory-tested by Fluidion in conjunction with the Paulsson optical fiber tool, and compatibility of the two technologies was demonstrated. The high-pressure high-temperature microscopy bench was used for the tests and was coupled to granite block and different types of attenuating foams. The Fluidion AME signal was clearly observable, with high SNR, whereas competing sensors (standard geophone and high-end accelerometer) were not able to detect the signal.

Process development was performed successfully for manufacturing full silicon devices (use of glass layers can now be eliminated in future designs). Initial devices for acoustic testing manufactured and tested successfully with Paulsson FOSS tool, as described above.

Hydrodynamic simulation of the timing circuit was performed by Fluidion under representative pressure and temperature conditions (Poiseuille flow simulation in rectangular channel geometry). Three timing circuit designs were cleanroom-manufactured out of silicon and glass, corresponding to 5min, 30min and 1hr timing delays. Tests using specific slick-water formulations are in preparation.

Multiple AME device designs were realized (4) by Fluidion, corresponding to different geometries (rod, cylinder, rectangular) and glass-silicon manufacturing process. FEM stress analysis was performed for expected hydrostatic and localized stress conditions.

All-silicon manufacturing was performed by Fluidion as well, using a recently-developed eutectic bonding process for sealing the devices. This manufacturing technique offers better machining capabilities for complex MEMS and microfluidic devices, since the etching of silicon is much better controlled than that of glass. Fluidion also developed a new brazing process for sealing the devices that requires the use of a pick-and-place machine. This type of manufacturing allows tremendous amount of customization, since the devices are built on a one-by-one basis (automation possible) and therefore are not constrained by the batch manufacturing process requirements. Using the above techniques, two generations of high-pressure AMEs were manufactured by Fluidion in the cleanroom, ensuring the compatibility of the new bonding process with the ensemble of fabrication steps required for AME integration.

Two additional manufacturing runs were made by Fluidion, which involved full silicon devices with large internal volume cavities (~10µL), and devices incorporating a fluidic clock, for initial testing. Fluidion also performed a manufacturing run in plastic material (PMMA for this test run), where the devices integrated an Aluminum timing membrane and a silicon separation membrane. The actuator for these devices was manufactured out of PMMA as well. PMMA was chosen for the ease of manufacturing, however, if successful, the process could then be adapted for higher temperature plastics such as PEEK. New designs of silicon separation membranes were manufactured as well, to optimize the collapse and their acoustic emission.

Tests with these devices were performed to qualify the clock functionality and the capability of the actuator to collapse the device at the end of the timing period. Tests were also performed in Paulsson Inc. facilities, in conjunction with the FOSVS tool. A new generation of devices was manufactured for this purpose, and testing was performed in a large water tank (swimming pool or equivalent). This allowed the tool and devices to be fully decoupled, thus allowing representative data to be obtained in laboratory-controlled conditions, as a pre-field experiment. For a more detailed description, see description of Task 12.0 results.

Paulsson has tested both the Fluidion and the Terves Injectable Micro Emitters in our laboratory. Favorable results have been achieved in term of ease of operation and projected cost to use these small silica sphere based devices.

The Terves IMEs will be significantly less expensive than the Fluidion AME devices but will have significantly lower acoustic energy per AME. This might be compensated by the large number of IAME/AME deployed using the Terves technology.

Task 3: Specification tasks for the vector sensor system.

Subtask 3.1: Paulsson, the Recipient, will develop the design for a Fiber Optic Seismic Vector Sensor robust enough to be field deployed into oil and gas wells and to meet and or exceed all environmental requirements of repeated use in wells drilled into shale formations. The requirements must include ability to withstand a temperature of up at least 500°F, a maximum pressure of 20,000 psi and withstand all fluids found in wells drilled in shale oil and gas fields

Achievement: We have designed a sensor that can operate to over 500°F, it has been tested to 608°F, a maximum pressure of over 20,000 psi and withstand all fluids found in wells drilled in shale oil and gas fields. A patent application has been filed and is currently processed by the USPTO.

Subtask 3.2: The Recipient will develop the design for a Fiber Optic Vector Sensor Array to meet and or exceed the geophysical requirements, such as sensitivity, bandwidth, vector fidelity set forth by shale oil and gas operators for use in oil and gas shale wells.

Achievement: We have reviewed the performance of our sensor with several operators of oil and gas shale wells. The operators we have discussed our sensor with have confirmed that our sensor meets their specification. Southwestern Energy decided to join this project after reviewing the design of our sensor system.

Subtask 3.3: The Recipient will develop the operational requirements for the interrogator system for the Vector Sensor system based on Institute of Electrical and Electronics Engineers (IEEE) standards. The Recipients will design an interrogator system that is technically able to handle a minimum of 1,000 channels per system while maintaining the data sensitivity of a minimum of 300 radians/g and a noise floor less than 50 ɳg/√Hz that are the necessary specifications to record high fidelity borehole seismic data.

Achievement: We will incorporate IEEE standards as appropriate. We have designed the interrogator to handle 1,000 channels while maintaining the data sensitivity of a minimum of 300 radians/g and a noise floor less than 50 ɳg/√Hz.

Subtask 3.4: The Recipient will develop the operational requirements for the pressure housing that will contain the fiber optic seismic vector sensors. The Recipient will design a pressure housing to protect the fiber optic seismic sensors from both the high-pressure environment and from the corrosive elements in the borehole fluids. The Recipient's designed specifications for the pressure housings are that they must survive a maximum pressure in the operator's oil and gas shale well of over 20,000 pounds per square inch (psi) at a temperature of 500°F.

Achievement: We have designed the pressure housing for the sensors that will survive a pressure in the operator's oil and gas shale well of over 20,000 pounds per square inch (psi) at a temperature of 500°F.

Task 4.0: Design of Fiber Optic Vector Sensor to allow operations at 500ºF

Subtask 4.1: The Recipient will design the Fiber Optic Vector Sensors.

The Recipient will select the fibers and the fiber coating to be used in the fiber optic seismic sensors that are suitable for vector sensors. The Recipient will select the fiber and fiber coating by working with a several fiber suppliers and by testing their fibers with the Recipient's sensor mandrels. The Recipient will select the fiber based on measured attenuation when the fiber is wrapped around the vector mandrels.

Achievement: We have selected the fibers and the fiber coatings to be used in the fiber optic seismic vector sensors. We have also verified that the fiber can be wrapped around the sensor without excessive attenuation of the laser light in the wrapped fiber.

The Recipient will select the design of the fiber optic seismic vector sensor. The Recipient will select the materials to be used for the fiber optic seismic sensors based on the materials ability of being hardened to a suitable hardness and stability with temperature and finally ability to be machined into the shape of the fiber optics sensors.

Achievement: We have selected the design of the fiber optic seismic vector sensor after reviewing more than 10 different designs. We have selected the material for the vector sensor mandrel which can be hardened to a suitable hardness and still be machined.

The Recipient will design the pressure housing for the Fiber Optic Vector Sensor array.

Achievement: We have designed the pressure housings for the fiber optic vector seismic sensors.

Subtask 4.2: The Recipient will select the material for the pressure housing with the following but not limited to criteria:

- The pressure rating of the pressure housing must be at least 20,000 psi and the temperature rating must be at least 500ºF.
- The pressure housing must withstand the environment found in injection and monitoring wells for long periods.
- The pressure housing must have a structural resonance over 2,000 Hertz (Hz).

Achievement: We have selected the material for the pressure housing for the fiber optic seismic sensors. We will be using Titanium and Inconel X750 for all the pressure pod components. The structural resonance is modeled to be about 2,500 Hz.

Subtask 4.3: The Recipient will design the fiber tube to optimize the acoustic coupling of the fiber in the tube for the Distributed Acoustic Sensors (DAS) array.

Achievement: We have designed a ¼" fiber tube with fiber that provides an outstanding response when used as a Distributed Acoustic Sensor (DAS).

The following are the minimum, but not limited to, steps to be completed by the Recipient during the design and optimization of the acoustic coupling of the fiber in the tube for the DAS array.

- The Recipient will assess how many fibers can be placed in a 0.150" OD Fiber-In-Metal-Tube (FIMT)
- The Recipient will complete test(s) to determine which fiber provides the highest Rayleigh Scattering response to seismic actuation
- The Recipient will complete test(s) to determine which fiber provides the best frequency response

Achievement: We have found that we can put in at least 15 fibers in a 15,000 ft. long fiber tube with an OD of 0.250”. We have tested several fibers and found one with the appropriate Rayleigh scattering response. We have also tested several fibers for frequency response and selected an appropriate fiber with the best combination of properties.

Subtask 4.4: The Recipient will design the sensor pod housing for operations at 500ºF.

The following are the minimum, but not limited to, steps to be completed by the Recipient during the design of the sensor pod housing for operations at 500ºF.

- The Recipient will assess which materials can withstand long term operation in oil and gas wells.
- The Recipient will assess which materials are strong enough to withstand the pressure in UOG wells
- The Recipient will assess the maximum weight for a field unit to be transportable
- The Recipient will assess the maximum size for a field unit to be transportable
- The Recipient will complete the design of a spool capable of being used as a field unit during the field operation
- The Recipient will complete the design of the power module for the field unit capable of operation during the field operation
- The Recipient will complete the design of the instrument room for the field unit to be used during the field operation.

Achievement: We have selected Titanium as the appropriate material for our sensor pods and Inconel for the fiber tube since can withstand long term operation in oil and gas wells. Titanium and Inconel are both strong enough to withstand the pressure in UOG wells. We have found that our field unit is light enough to be transported on a large truck. The field unit will fit inside a 20 ft. container that can be transported by a truck. We have designed a spool that fit inside a 20 ft. container. The 20 ft. container is big enough to house both the spool and the instrument room used during the field operations.

Decision Point: Fiber Optic Vector Sensors Design Acceptance

Achievement: We submitted the Go-No-go report to DOE on February 28, 2015 and received approval to proceed based on the technical progress we have made on the design of the Fiber Optic Vector Sensors. The Recipient provided the DOE TPO with a technical briefing.

Task 5.0: Manufacturing of the Fiber Optic Vector Sensors

Subtask 5.1: The Recipient will manufacture the Fiber Optic Vector Sensors.

Using the selected design of the fiber optic seismic sensor from Task 4 the Recipient will manufacture the first unit.

The following are the minimum, but not limited to, steps the Recipient will complete to manufacture the first unit.

- The recipient will select the material for the sensor based on a suitability as a mandrel material and stability as function of temperature
- The recipient will evaluate the machining process of the sensors based on discussion with machine shops and costs.
- The recipient will issue a request for quotes (RFQ) for the sensors
- The recipient will evaluate the submitted RFQ and select a machine shop for the sensors based on but not limited to the following selection process and criteria;

    - Ability to deliver in time
    - Price
    - Quality
    - Experience

The Recipient will manufacture a minimum of 300 fiber optic seismic vector sensors to be used in testing in the prototype and the survey systems.

The Recipient will test each sensor for consistency and uniformity in output using, but not limited, to the following test procedure.

- Measure the output of each sensor in radians/g using a low frequency tester
- Measure the cross-axis isolation in Decibel (dB) using a shaker table
- Test the sensor response at 392°F using a shaker table installed in an oven
- Test the sensor response after sensors are installed in the sensor pods

Subtask 5.2: The Recipient will test the existing interrogator with the fiber optic vector sensors.

The Recipient will develop design and test criteria for the interrogator for the fiber optic seismic sensors. Using the design criteria and the selected fiber test the Recipient will test the interrogator for the fiber optic seismic vector sensors.

The following testing procedure will be used during the testing of the interrogator for the fiber optic seismic vector sensors.

- Measure the system noise of the interrogator in ɳg/√Hz. This noise must be less than 50 ɳg/√Hz
- Measure the signal to noise ratio of the data recorded using the array interrogator. The Signal to Noise (S/N) ratio must be 50 times the S/N of standard geophones.
- Compare the data from the array interrogator with the data through a single channel interrogator

Achievement: We selected the material for the sensors and we have completed the machining of the 330 sensors in our in-house machine shop and at a specialty EDM machine shops. We determined that the combination of using our in-house machine shop and a specialty EDM machine shops allowed us to maintain the best quality at a reasonable price and in the same time keep a predictable manufacturing schedule.

We have performed extensive QC testing of the sensors and most have passed our QC process.

We have tested the existing interrogator with the fiber optic vector sensors and developed a process to reduce the optical noise in the interrogator to improve the S/N ratio of the data recorded by the sensor by a factor of 10. This task in 100% complete.

Task 6.0: Environmental and Bench Test of Fiber Optic Vector Sensor Assemblies

Subtask 6.1: The Recipient will test the Fiber Optic Vector Sensors

The Recipient will conduct performance test the Fiber Optic 3C Vector Sensor assemblies on a shaker table at temperatures ranging from 77ºF - 392ºF in 45ºF steps at frequencies ranging from 5 to 2,000 Hz

The Recipient will test the performance of the Fiber Optic Vector Sensor using, but not limited to, the following set of tests

The Recipient will conduct the following shaker table procedure;

- Shake the 3C assemblies and measure the output of the three sensors
- Measure amplitude of the 3C sensors
- Measure the frequency response of the 3C sensors
- Assess the cross-axis isolation between the sensors

The Recipient will test the Fiber Optic Vector Sensors at temperature ranging from 77ºF - 392ºF in 45ºF steps using tap tests.

- Tap at the axis of each of the three sensors
- Measure amplitude of the 3C sensors
- Measure the frequency response of the 3C sensors

The Recipient will compare the results of the tests with the Fiber Optic Vector Sensor and interrogator with test results using an industry standard borehole seismic high temperature geophone.

Sub Task 6.2: The Recipient will test the existing interrogator system with the fiber optic seismic vector sensors.

The interrogator test will be conducted by the Recipient using the vector sensors. The Interrogator test using the vector sensors consists of but is not limited to the following steps;

- The interrogator test conducted by the Recipient will determine the noise floor of the system in ɳg/√Hz.
- The interrogator test conducted by the Recipient will determine the sensitivity of the

system in radians/g.

Subtask 6.3: The Recipient will test the Fiber Optic Vector Sensor pod and associated fiber optic terminations.

The Recipient will test the Fiber Optic Vector Sensor array pod, DAS in tubing and termination from 77 - 500ºF in 45ºF steps at 20,000 psi.

The Recipient will test the Fiber Optic Vector Sensor array pod, DAS in tubing and termination using, but not limited to, the following detailed test procedure.

- Tap test at pressures of 15 – 20,000 psi in 2,000 psi steps
- Tap test at temperature of 77°F – 500°F in 45°F steps

Achievement: We have completed all the laboratory tests listed in Task 6 including testing of sensors inside pod housings to a pressure of 3,000 psi. We have tested sensors to over 600°F at frequencies ranging from 0.03 Hz to 6,000 using both shaker tables and tap tests. The sensors have shown to meet or exceed all specifications such as being about 100 times as sensitive as geophones at higher frequencies, able to operate at 600°F, able to record broad band data from 0.03 – 6,000 Hz and with an outstanding vector fidelity. We have expanded the testing to include fibers with different coatings.

The sensor pods are scheduled to be manufactured in BP2 – BP3 so all testing combining sensors and sensor pods will by necessity occur after the sensor pods have been completed.

We have tested the fiber optic sensors with the existing interrogator system and we are using the test data to upgrade and fine tune the design of our new interrogator that will serve the 100 level 3C system. This task is 100% complete except for the test at 20,000 psi. We have however modeled the pressure performance and found that the sensor pod design can handle 30,000 psi.

Task 7.0: Borehole Seismic System Performance Analysis

Subtask 7.1: The Recipient will generate a comprehensive analysis of the performance of the Phase 1 Fiber Optic Vector Sensors and determine the probability that the components can meet the performance requirements and operational conditions specified.

Subtask 7.2: The Recipient will analyze all the data collected and compare the data from the fiber optic seismic vector sensors with data from geophones and accelerometers.

Achievement: We have performed an extensive set of measurements on the fiber optic vector sensors. The sensors perform as expected with a sensitivity of more than 400 Radians/g. The sensors have also proved to be able record data from 0.03 Hz to 6,000 Hz. We have compared the data from the fiber optic seismic sensors geophones and accelerometer. In a side by side laboratory test of the fiber optic seismic sensors with geophones and accelerometers using the Acoustic Micro Emitters (AME's) as the seismic source the fiber optic seismic sensor produced data with a S/N ratio of 250, the piezo electric accelerometers produced data with a

S/N ratio of five while the geophones did not record any detectable data. This task is 100% completed.

The developed seismic sensors will record outstanding seismic data in the field system once completed. We have also been tuning the fiber optic interferometric interrogator to maximize the signal to noise ratio of the data recorded with our sensors.

Budget Period II

Task 8.0: The Recipient will develop a real-time processing interface

A real time processing interface for Seismic Imaging and Micro Seismic Mapping software working together with the optical interrogator will allow processing in real time.

Subtask 8.1: The output of the optical interrogator will be tailored to be suitable to be converted to SEGY and SEG2 data formats

Subtask 8.2: Real time software will be written to allow the output from the optical interrogator to be converted to SEGY and SEG2 in real time.

Subtask 8.3: The current Recipient micro seismic monitoring software will be converted to a real-time monitoring system

Subtask 8.4: Display software will be developed that will allow the real time to display of the micro seismic events recorded and analyzed by the real-time mapping system

Achievement: The Recipient has developed a real-time interface for the seismic imaging and micro seismic monitoring. The output of the optical interrogator is converted in real time to SEGY or SEG2 format. The Recipient has developed real time display software and using this software the Recipient can process, display and analyze the data in real time. This task is 100% complete.

Task 9.0: The Recipient will manufacture a minimum of two prototypes of the 2” OD pressure pods for the fiber optic vector sensor.

Subtask 9.1: The Recipient will manufacture the first units of the 2” OD pressure pods for the Fiber Optic Vector Sensor array using, but not limited to, the following manufacturing procedures.

- The design of the pressure pods will be reviewed on the Solid Works Software or equivalent software package.
- After analysis, the prototypes will be printed in 3D
- An analysis of the 3D printed prototypes will be completed.
- After successful analysis of the 3D printed prototypes machine the first article of the 2” OD pressure pods.

Subtask 9.2: The Recipient will manufacture the first units of the fiber optic terminations using, but not limited to, the following manufacturing procedure;

- The design of the fiber optic terminations will be reviewed on the Solid Works Software
- After analysis of the Solid Works the prototypes will be printed in 3D
- Analyze the 3D printed prototypes.
- After successful analysis of the 3D printed prototypes machine the first article of the fiber optic terminations.

Decision Point: Review and acceptance of the final sensor pod design prior to manufacturing the 100 Units as described in Task 10.0 and beyond.

Achievement: We submitted the Go-No-go report to DOE on April 14, 2016 and received approval to proceed based on the technical progress we have made on the design and prototyping of the pod for the Fiber Optic Vector Sensors. The Recipient provided the DOE TPO with a technical briefing. The task is 100% complete.

Task 10.0: Manufacture 100 units of the sensor pods for the vector sensors

The sensor pods are pressure vessels that protect the sensors from pressure and the well fluids. The Recipient will manufacture a minimum of 100 units of the 2” OD sensor pods for the fiber optic vector sensor array.

- The Recipient will submit the Recipient designed drawings of the sensor pod components to at least three, but not limited to, machine shops for quotes and delivery schedules
- The Recipient will select the most advantageous offer for machining the components for the sensor pods based on
  - Quoted cost
  - Quoted Delivery Schedule
  - Manufacturing Capabilities
  - Experience with pressure vessels operating at pressures over 20,000 psi
  - Experience machining Inconel materials.
- After delivery of the components, the Recipient will perform the following acceptance tests:
  - Assemble the components to complete pods
  - Label all pod components as a set using laser engraving
  - Measure all critical dimensions of the received components
  - Document all measured dimension in the pod specific traveler
  - Issue an acceptance document to the manufacturer

Achievement: We submitted the design to three outside machine shops. The quotes received were high and the delivery schedule long and uncertain. The Recipient therefor purchased one large CNC mill and two large CNC lathes to meet the machining need for our DOE program. Paulsson, The Recipient, now have two CNC mills, one large and one small, and three CNC lathes, two large and one small. We have completed the programming for all the components and completed the first item of the metal components. 100% of the material has been purchased and the machining task will be 100% completed by December 18, 2016.

Task 11: Assemble a 100-level lab test system of the Fiber Optic Vector Sensors

Subtask 11.1: The Recipient will assemble the Fiber Optic Vector Sensor array from the manufactured components for lab analysis.

Subtask 11.2: The Recipient will test the Fiber Optic Vector Sensor array at the Recipients Facility for performance and uniform output during the assembly using optical instrumentation.

Achievement: We have fully assembled a 16 level array. We have performed many tests on a sub array with outstanding results. The sensors tested have a consistent output. We have shown that we can record high fidelity vector seismic data with a Moment Magnitude of smaller than M-5.0. We have also shown that we can record high frequency laboratory data from an event with an energy level of 2.5 µJ which is the equivalent of smaller than M-7.0. This task is 100% complete as far as the sensor go.

Budget Period III – Manufacturing of AME's and sensor pod housings

PLEASE NOTE: The report for task 12 will also be valid for Budget Period III since it is a multi-year task.

Task 12.0: Laboratory testing and design optimization of Acoustic Micro Emitters (AME's)

Subtask 12.1: AME laboratory testing and design optimization

Fluidion will cleanroom fabricate a minimum of four different versions per the AME's designed during Task 2.1, in quantities of minimum 100 AME's each for use in the characterization study. Fluidion will perform regular quality checks at critical fabrication stages and at final device delivery.

The laboratory testing of the devices conducted by Fluidion will follow the same protocol for a minimum of four AME design types, and will consist of three types of tests listed in the following tasks:

Fludion will conduct Laboratory Pressure Testing on microscopy bench, at a minimum with simultaneous video microscopy and acoustic signal recording.

Fludion will perform this test at a minimum using a combination of custom-designed and general laboratory equipment.

Fludion will verify the acoustic emission using at a minimum, laboratory-scale sensors, and will record the specific source term of different device designs, effect of pressure and shape on the generated signal.

Fludion will conduct Laboratory Fluid Transport using at a minimum but not limited to, flow test fixtures and different sizes of AME's per the specifications developed in Task 2.1, to gain a better understanding of the transport properties of different types of AME's in simulated hydraulic fractures. Tests of proppant and AME transport and settling in simulated fractures from 0.5 to 10 mm in width will be performed and the proppant settling pattern and the AME distribution within the proppant pack will be measured using as a minimum camera recordings and particle analysis software.

Paulsson will conduct the Pressure Tank Test using the fiber optic seismic vector sensor to enable recording of the selected AME's acoustic signatures in simulated in-situ conditions.

Paulsson will use the high-sensitivity fiber-optic seismic tool placed in the same high-pressure high-temperature test vessel simulating the well, as the AME's.

Paulsson will record the source term using the same tool and electronics that will be used in the final field test, and will provide estimates about ultimate signal detectability, thus Paulsson will provide an estimate of the maximum possible recording distance for optimizing the placement of the off-set well for recording during the field test.

Fluidion will incorporate feedback and data from the different experiments performed above and will complete design of an optimized AME geometry that will be used in the field tests.

Achievement:

Fluidion designed and fabricated a proppant flow fixture from PMMA using laser cutting as well as 3D laser engraving. An actual fracture geometry was used (Hurst exponent: 0.8) and could be imprinted onto the fracture walls using laser ablation technology to simulate a real environment. A literature review was performed of all proppant transport and fracture geometry literature to date, and previous experiments were analyzed, and conditions adapted to the current setup. Transparent glass microspheres were used to allow the visualization of proppant, in conjunction with both opaque and photo-luminescent mockup AMEs. We used widths from 1 to 12 mm, which are representative of actual fracture geometries recorded in the field. We also included tapered joints to allow for variable width fractures (6mm to 2mm and 3mm to 1mm). Different geometries of AME (cylindrical vs. rectangular, different sizes) were attempted, and the time-dependent distribution of proppant and AMEs was recorded using camera photos and movies. Test were performed in both smooth and rough geometries with different AME geometries (cylindrical, rectangular, rod). The experiments using this setup are ongoing. Significant amounts of data have been collected and are currently being processed. The data obtained so far demonstrates that:

1. AME particles become wedged wherever fracture width is equal to the smallest dimension of the AME, thus allowing to obtain information about fracture directions and width
2. Cylindrical and rectangular AMEs show similar strength and transport characteristics
3. AME particles can dynamically follow fracture development (they move location in real time as a consequence of fracture widening)

4. Different size AMEs can be combined and will stop at different places in the fracture, providing information about fracture width gradient, aperture vs. radial distance, and hence total fracture extent.

A new manufacturing run for devices incorporating fluidic clocks for delaying the acoustic emission is being prepared and should be initiated at the end of August 2016. The manufacturing run will incorporate all past device design evolutions that have been validated and should result in a batch of device that will allow realistic testing with the FOSS tool. The devices will utilize the novel silicon bonding process developed in this project, which should allow complex MEMS device geometries to be manufactured in batch processes.

Initial testing of the signal emission from the August batch will be performed in the Fluidion high-pressure facilities (microscopy bench as well as large-scale hyperbaric pressure tank) and will allow independent recording of the acoustic source term. The functional AME testing was performed in September 2016.

Additional testing was performed in September 2016 in conjunction with the FOSS tool, on the premises of Paulsson Inc. The testing proved that manufacturing of the August batch was successful, and that the AME signal could be recorded using the actual field sensor, thus providing increased confidence in a successful initial field test. Testing consisted of placing different quantities of AMEs of different types in a miniaturized high-pressure cavity, which could be pressurized using a manual hydraulic pump. The high-pressure cavity was lowered in a swimming pool. A low-cost hydrophone was in direct contact with the cavity, allowing measurement of the source term. A certain distance away (7-20ft) were placed different acoustic sensors (FOSS, geophone, fiber optic hydrophone), which recorded the transmitted signal in parallel. The results proved that excellent signal to noise ratio can be recorded with the FOSVS and fiber optic hydrophone, with the majority of the acoustic energy at frequencies below 5kHz. The geophone, on the other hand, had a hard time picking up the signal, which is probably due to the lack of sensitivity at high frequencies.

A new batch of high-pressure AMEs was cleanroom-manufactured in October 2016, using a full silicon construction and a proprietary silicon bonding process. These AMEs will collapse between 4000 and 7000 psi, ensuring field survivability as well as significant acoustic signal release. Devices with modified membranes were also manufactured, which should allow rapid collapse therefore increasing acoustic output. The devices will be tested in a larger acoustic medium environment, such as a lake or large pool, and should also allow testing in an initial well.

The cleanroom being temporarily closed for relocation into new, larger facility, work since November 2016 has mostly focused on design, finite element analysis and optimization of new AME designs. The fabrication process has been streamlined to allow rapid fabrication of larger batches, and specifically for enabling complete collapse of the devices (with maximal energy release) as compared to partial collapse.

Different adhesive bonding techniques have been developed and tested, to allow full-strength structural integrity yet enable a variation in the materials used for fabrication (such as use of ceramics and laser-machined plastic and metal parts). Epoxide and UV-curable adhesives have been tested under different temperature and pressure conditions. Use of hybrid materials will allow for better resilience and fracture resistance of the AME body yet allow for strong acoustic emission upon collapse of the silicon cap. Tests of such new devices have been performed in a laboratory pressure cell under the inverted microscope.

Fluidion completed the designs for smaller devices and the fabrication process was validated with the cleanroom engineering support team. The production of new batches is setup to start as soon as new cleanroom facility reopens.

Fluidion initiated the design of an AME injection device to allow direct injection under high pressure conditions, past the fracking pump trucks. This will allow bypass of the pumps, which will ensure 100% survival of all the AMEs to the wellhead and into the wellbore being fractured. This should greatly improve the throughput and allow larger number of micro-acoustic events.

In parallel, preparations for a new lake test with the Paulsson FOSS tool was completed. A batch of larger (4mm) AMEs was provided to Paulsson for initial testing of the pod pressure vessel and have been successfully tested. Additional batches were provided and/or manufactured, as needed, for future tests including an initial well test.

Since January 2017, the Fluidion work has concentrated on two aspects:

1. Testing new designs for integrating passive timing into field-worthy devices
2. Manufacturing test devices for supporting on-going validation and field tests

Fluidion manufactured several batches of AME’s in different configurations and supplied Paulsson Inc. with testing devices to perform field testing at lake location. We optimized 4mm cavity shape for maximum emission at 4000 and 8000 psi collapse pressures. Successful detection could thus be achieved at distances of over 100ft, in noisy environment. It is therefore likely that detection at significantly longer distances could be obtained in quiet environment, and with direct AME-rock coupling.

Fluidion prepared a new AME manufacturing plan and are provisioning for a field test in a test well. We have been performing laboratory testing of trial devices integrating fluidic passive clocks, but with mixed results so far.

Task 13.0: Write the report on system developed, data recorded and processed

The Recipient will develop a report that includes but not limited to the following information; Description of the developed system, data recorded during the developed system testing, processed data and conclusion.

Achievement: We have completed the PPT report on the system developed, data recorded and processed. This report was completed September 30, 2016. The team presented an update to DOE in March 2017. An updated report was completed by September 30, 2017.

Task 14.0: Final AME design, testing and manufacturing –

Sub Task 14.1 by Fluidion. The Recipient will integrate lessons and results from tests performed in previous task to manufacture a highly-optimized final design of the AMEs that will be used in the actual field test

Fluidion fabricated the AME's per the final design completed in Task 2.2 and the fabrication will be completed in a certified cleanroom Micro-Electro-Mechanical System (MEMS) fabrication capabilities and equipment, class 10000 minimum in general areas and 1000 minimum in lithography areas).

Pressure tank testing was performed on the final design as well, to ensure detectability and to calibrate the seismic tool array.

Sub Task 14.1. Injectable Micro Emitters by Terves, LLC.

Paulsson has identified an alternative source for very small, smaller than 100 um, Injectable Micro Emitters (IMEs). The size of these IMEs match the size of the 40-70 and 100 mesh proppant. The small and proppant matching size should make these Micro Emitters much easier to inject together with the proppant. These IME's are based on small silica spheres with a dissolvable coating. These IME are provided by Terves LLC located in Euclid, Ohio.

Achievement: Paulsson has successfully tested the small IME's in the laboratory and in small field tests. Paulsson introduced the Terves IME to the Marcellus Shale project and they purchased 12,000 lbs. of the Terves IME's to be tested during fracturing of the Marcellus Shale.

Task 15.0: The Recipient upgrade of the Interrogator

The Recipient will upgrade the interrogator for the Fiber Optic Vector Sensors. Using the borehole seismic system performance criteria manufacture the optical interrogator for the fiber optic seismic vector sensor array.

Subtask 15.1: The Recipient will manufacture the interrogator to meet the following performance criteria:

- Interrogator noise floor: <50ng $Hz^{-1/2}$; <250ng over 10-2,000 Hz band
- System bandwidth: 0 – 6,000 Hz
- Digitized sample size: 32 bit or greater
- Dynamic range: 180dB
- Sample bit resolution/sensitivity: 3ng/bit
- Output formats: SEGY and SEG2

Achievement: We have upgraded and tested the optical interrogator. We have confirmed that we can record data up to 10,000 Hz with the upgraded interrogator. We have improved the sensitivity of the sensor by refining our manufacturing processes. We are currently working on expanding and improving the dynamic range of the interrogator including modifying the telemetry. We are in the process to upgrade the electronics design to lower the low frequency noise. We have successfully reduced the instrument noise of the interrogator.

Task 16.0: The Recipient will manufacture the sensor pod housing components for UOG field service for the 100 level 3C fiber optic vector sensor

The Recipient will assemble the sensor pod housings using, but not limited to the following steps;

- Machine the components for the sensor pod housings
- Weld the tool joints to the main body of the sensor pod housings
- Install the clamping mechanism

Achievement: At the end of the project we are still in the process of machining the components for the sensor pod housings. We expect to complete this process in FY2020.

Task 17.0: The Recipient will test all components of the fiber optic vector sensor system

Subtask 17.1: The Recipient will test Fiber Optic Vector Sensors for data consistency and data uniformity using the developed interrogator

Subtask 17.2: The Recipient will test the interrogator capable of operating a 300 channel Fiber Optic Vector Sensor array using the Recipients laboratory test facility

Subtask 17.3: The Recipient will test the 2” pressure housings for the fiber optic seismic sensors and fiber optic connectors using a commercial pressure vessel.

Achievement: We concluded a lake test of the AME and FOSVS combination to establish the detectable range of the AME’s in an acoustic medium. We easily detected the AME data at a distance of over 100 ft despite severe environmental noise in the lake. The lake was very noisy for reasons that are not clear, but it might be due to the hydroelectric power station that is part of the lake. The lake environment had 50 – 100 times higher noise than the pool environment used for one of the tests. In a normal lake environment, we will be able to see the data from the AME’s at a distance over 500 ft.

Budget Period IV - Field test of the developed micro seismic monitoring system and the AME’s.

Task 18.0: Assemble 100 level 3C Fiber Optic Vector Sensor array

Subtask 18.1: The Recipient will assemble the Fiber Optic Vector Sensor array from the manufactured components.

Subtask 18.2: The Recipient will spool the Fiber Optic Vector Sensor array onto the field spool.

Subtask 18.3: The Recipient will test the Fiber Optic Vector Sensor array at the test facility prior to deployment in a well using laboratory shaker equipment

Achievement: We have started to do test assemblies of the Fiber Optic Seismic Vector Sensor array. This process is taking longer than expected. We have started to plan for a survey with an UOG operator. This test survey will likely take place in FY2020.

Task 19.0: Borehole seismic survey using the 100 level 3C Fiber Optic Vector Sensor array

Subtask 19.1: The Recipient will develop Field Test Parameters Report for the field test to include the following

1. Time line
2. List of personnel involved
3. Site description
4. Well description
5. List of data acquisition objectives
6. Description of tools and techniques to be deployed

The work plan will be vetted by both DOE and the field operator. A mutually agreed upon plan shall be finalized prior to proceeding to subtask 19.3

Subtask 19.2: The Recipient will develop a safety plan for the survey including

1. Names of safety personnel
2. List of potential hazards
3. Contact numbers for local health care facilities including maps to the nearest hospital and emergency room

Subtask 19.3: Per the approved plans developed during subtasks 19.1 &19.2, the Recipient will deploy a 100 level Fiber Optic Vector Sensor array in one vertical or horizontal well deployed on 15,000 ft. of tubing. This test will be performed in a well provided by designated operator. The well that will be used for this test will be selected in consultation with the Operator and DOE project manager.

Subtask 19.4: The Recipient will perform a 3D VSP survey to get sufficient data to build a seismic 3D image and a 3D velocity model

Subtask 19.5 The Recipient will perform a micro seismic survey using both vector sensors and DAS array during the fracking operation

Subtask 19.6 The Recipient will perform a micro seismic survey using both vector sensors and DAS array after the fracking operator to listen to the acoustic data from the AME’s.

Achievement: This task was replaced by the survey performed for Battelle which showed that the FOSVS technology was able to track fluid flow and micro seismic generated by a very small, less than 300 psi, increase in the pore pressure due to the injection of CO_2 into a fractured rock.

Task 20.0: Processing of the recorded Micro Seismic and 3D Borehole Seismic Technology (BST) and Vertical Seismic Profile (VSP) data

Subtask 20.1: The Recipient will build velocity model and migrate the 3D VSP data using Kirchhoff Depth Migration, Generalized Interferometric Migration, Reverse Time Migration and Multiples Migration software.

Subtask 20.2: The Recipient will map fractures and faults using Generalized Interferometric Imaging. Map the Micro Seismic Events recorded in 3D using Recipients software or equivalent. The Recipient will map the location of the proppants based on micro seismic data.

Subtask 20.3: The Recipient will interpret the data.
During this subtask the Recipient will integrate the geologic model and the reservoir model developed by the operator with the results from monitoring the micro seismic data recorded during the fracturing stage. The micro seismic data the fracturing stage will then be compared with the data recorded from the AME's.

Achievement: Paulsson developed a velocity model for the Battelle survey. The active seismic source data was not of sufficient quantity nor quality to generate a 3D Image.

In 2019 Paulsson did a series of calibrated laboratory tests comparing the micro seismic events from the Terves Injectable Acoustic Micro Emitters (IAME) with calibrated sources.

Impact Tests

From the calculations the potential energy of the uncoated VisionFrax at 8,000 psi range from 0.122 µJ to 5.782 µJ for a normal particle size distribution.

We selected the following metal spheres for the impact tests to compare the amplitude recorded for comparable potential energy.

- 304 stainless steel, 1/16" diameter, 16.7 mg, McMaster PN: 9291K41
- 2017 aluminum alloy, 3/32" diameter, 20.1 mg, McMaster PN: 34665K27

Spheres dropped from 21.4 mm height have the following calculated potential energy.

Sphere Material	McMaster PN	Sphere Mass (mg)	PE (uJ)
304 stainless steel	9291K41	16.7	**3.50**
2017 aluminum alloy	34665K27	20.1	**4.22**

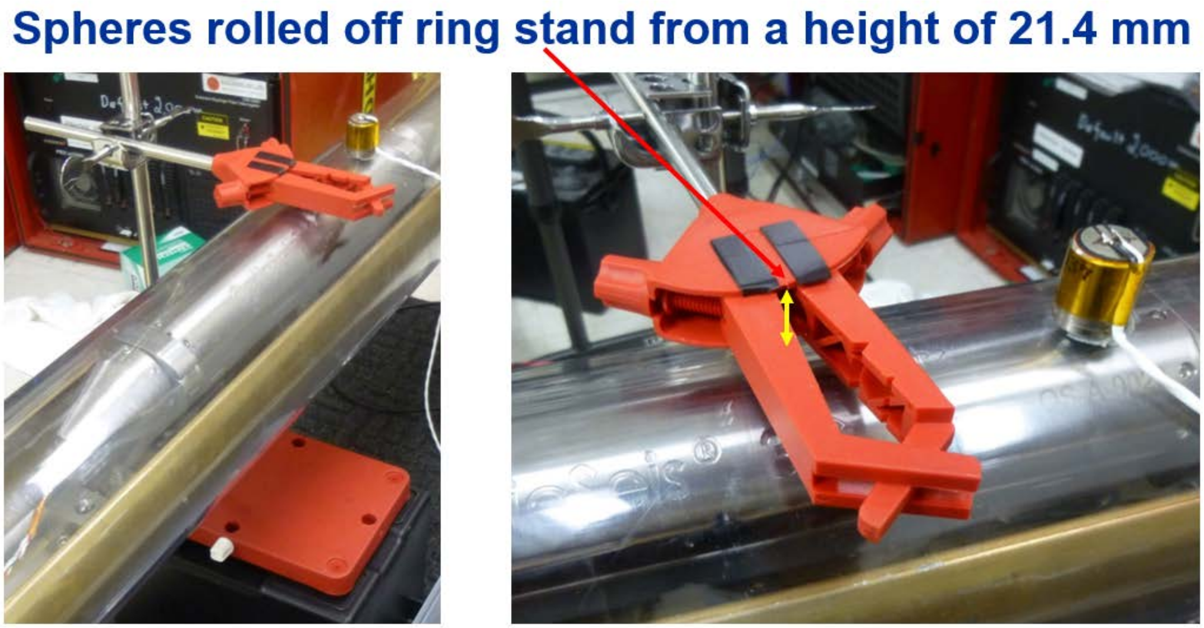

Figure 7. Test Setup – Impact Tests with several calibrated weight drops

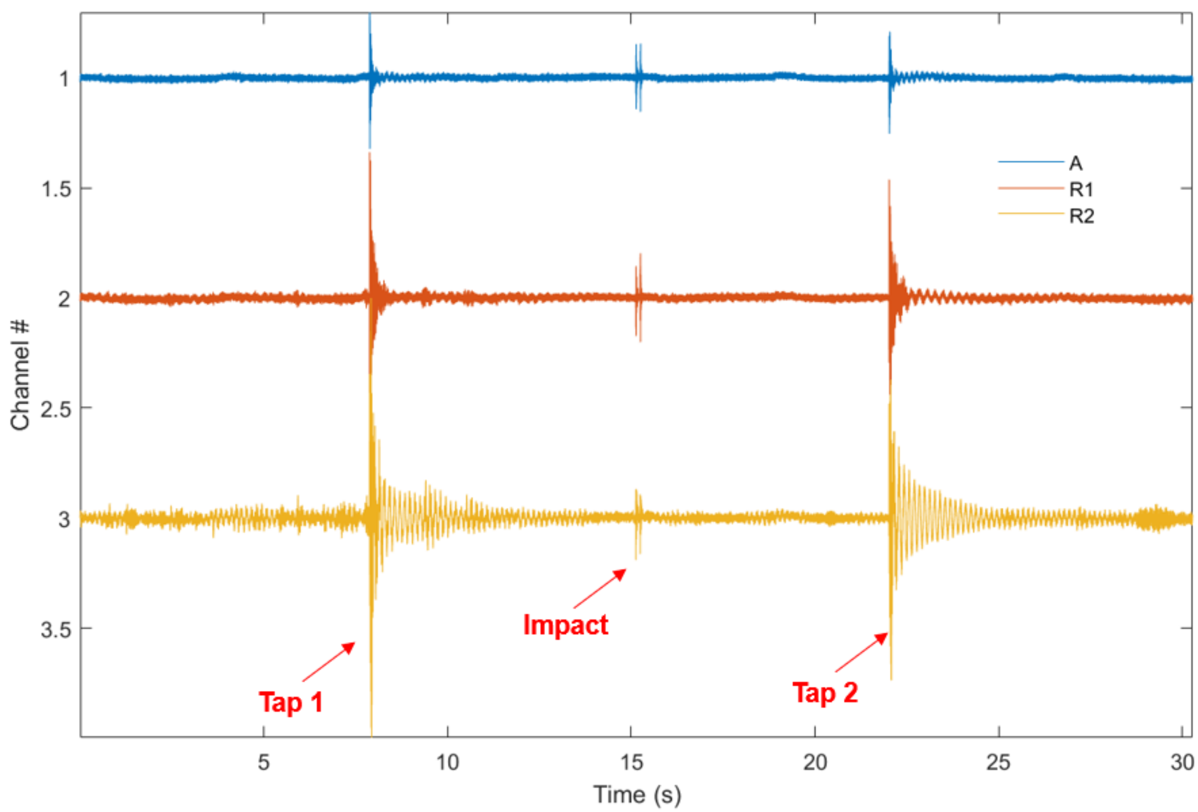

Figure 8. FOSVS Data: 304 SS Bearing Impact, 1/16” Diam., 16.7mg, 21.4mm Height providing a potential energy of 3.5 μJ.

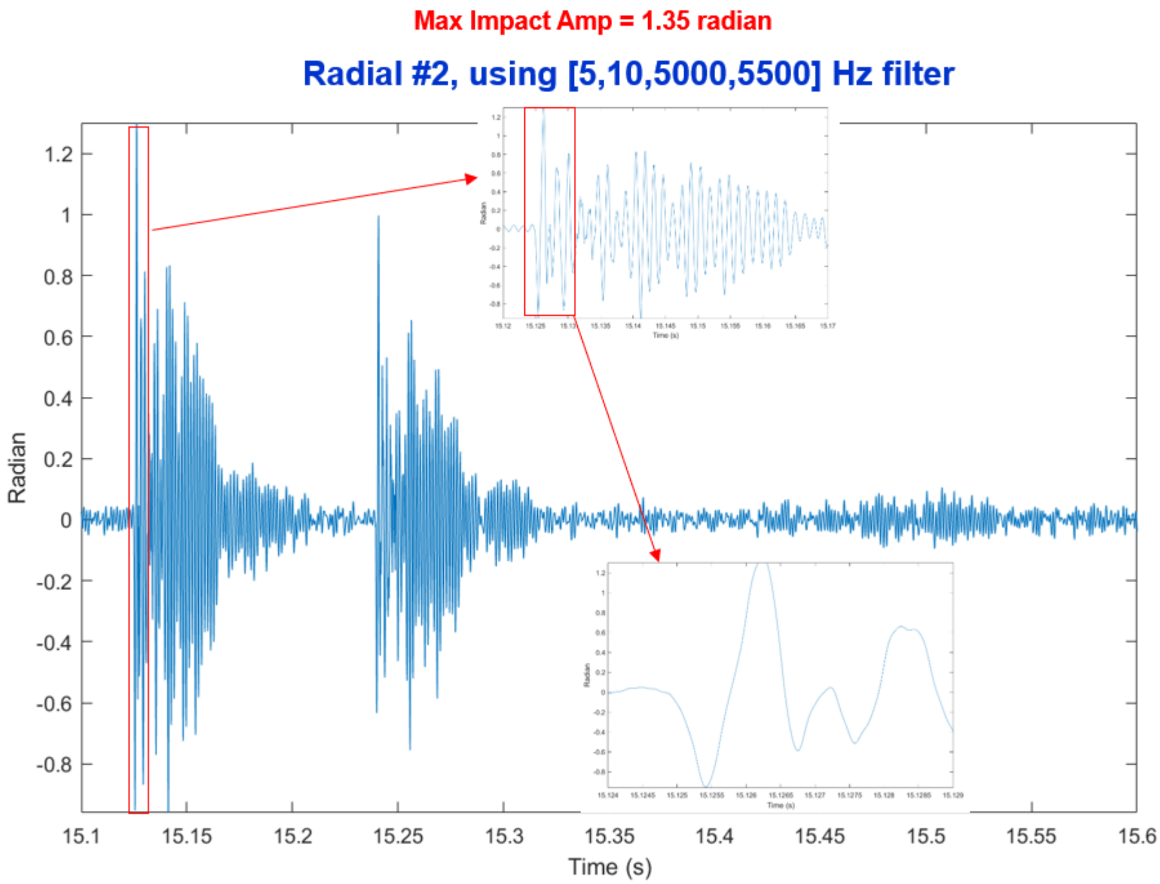

Figure 9. Zoom in on FOSVS Data: 304 SS Bearing Impact, 1/16” Diam., 16.7mg, 21.4mm Height providing a potential energy of 3.5 μJ.

Figures 7 to 11 show data from calibrated tests of weight drop on to the pod with FOSVS sensors. The tests are meant to emulate the result from using the Terves Injectable Acoustic Micro Emitters (IAME).

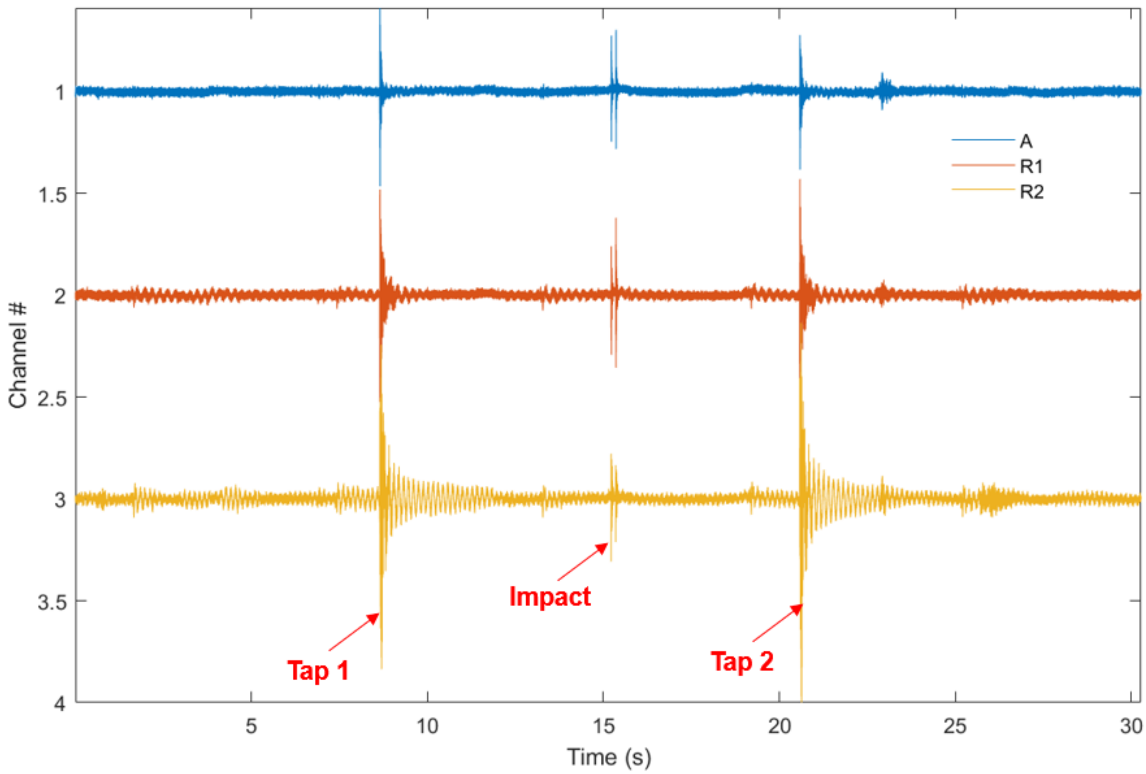

Figure 10. FOSVS Data from taps and calibrated weight drops: 2017 Al. Bearing Impact, 3/32" Diam., 20.1 mg, 21.4mm Height providing a potential energy of 4.22 µJ.

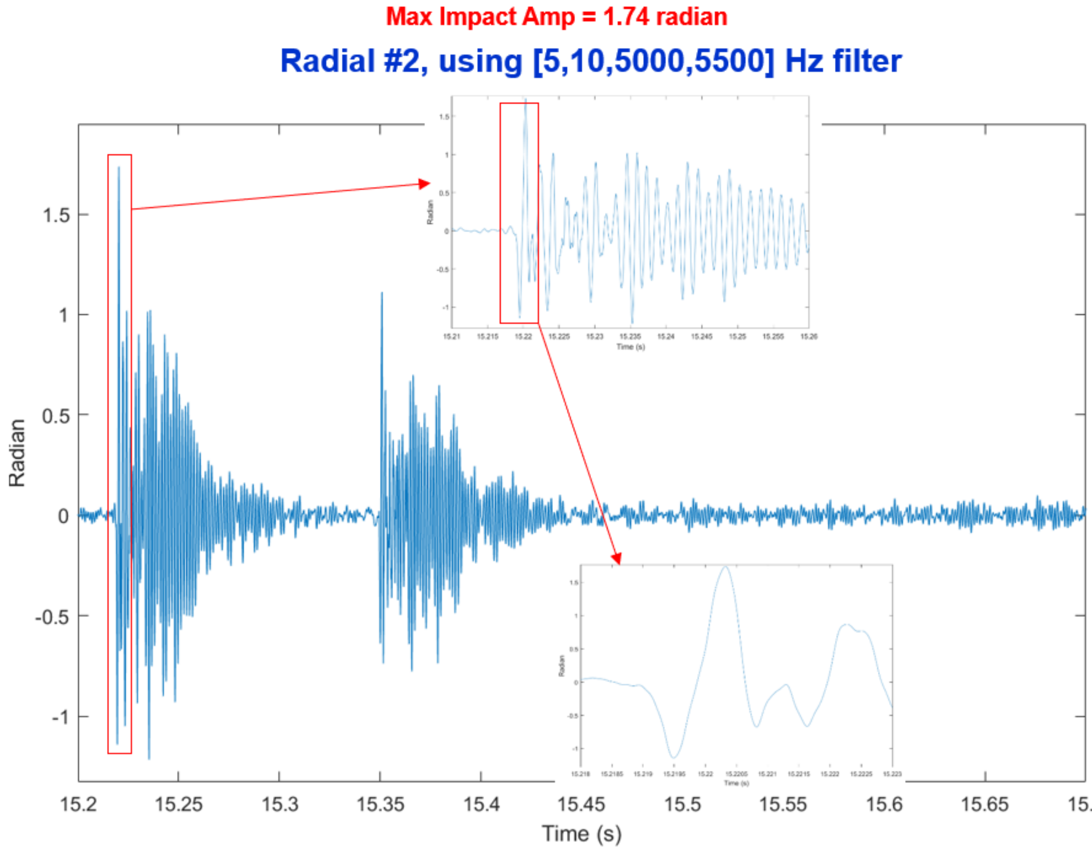

Figure 11. Zoom in on FOSVS Data in previous Figure: 2017 Al. Bearing Impact, 3/32" Diam., 20.1 mg, 21.4mm Height providing a potential energy of 4.22 μJ.

OBSERVATIONS AND CONCLUSIONS

1. We have successfully developed all the components for injecting and monitoring Injectable Acoustic Micro Emitters into hydro fractures by mixing the IAME with proppant in small concentrations.
2. We have verified the operation and function in calibrated laboratory tests.
3. The field test was cancelled by the operator for economic and operational reasons. The UOG oil field that was originally planned was sold by the operator South Westernenergy (SWN)
4. During the laboratory tests the measured impact amplitude of the dropped solid metal bearings with higher potential energy was proportionally higher. This indicates that higher measured amplitudes from the collapse of the Acoustic Micro Emitters is likely from higher potential energy. This is due to either higher collapse pressure or larger internal volume.
5. While the Cenosphere samples are much bigger (from magnification images), the hollow glass spheres provide more energy.
6. We matched the amplitudes of the waveforms of the Acoustic Micro Emitters (Acoustic Micro Emitters) analyzed to the amplitudes of the dropped solid metal bearings.
7. Amplitudes from the Acoustic Micro Emitters is comparable to those from the drops of the solid metal bearings. This indicates that the potential energy is also comparable and that the calculations are reasonable.

CHANGES/PROBLEMS:

Changes in approach and reasons for change

We are studying how to improve the low frequency response of the optical seismic sensor system. The current optical interrogator has significant noise at lower frequencies. We need to lower the low frequency noise and increase the dynamic range of the interrogator. We are in the process to build a low noise optical interrogator.

We experimented using fibers with different coatings to improve the S/N ratio of the sensors. Since we did not achieve any improvements we will continue to use the polyimide coated fibers for our sensors.

Actual or anticipated problems or delays and actions or plans to resolve them

The manufacturing of components is taking much longer time than anticipated. To speed up the manufacturing we purchased a large CNC Mill in December 2015, a large CNC lathe with milling capabilities in May 2016 and a second large CNC lathe with milling capabilities in September 2016. Paulsson now has two CNC mills and three CNC lathes. After completing the programming of the CNC machines, the manufacturing has become very efficient. The machining of all components will be completed in FY2020. Paulsson has purchased all the metals for the machining the system. The costs to complete the system includes the cost to test the system in the laboratory.

A test at a test site is planned for FY2020.

Changes that have a significant impact on expenditures

Manufacturing of the borehole seismic system is taking much longer than expected. Paulsson requested an NCE until 9/30/2019. The request for the NCE was approved.

SPECIAL REPORTING REQUIRMENTS:

Two significant changes occurred during the project.

The operating company, Southwestern Energy (SWN), that initially offered to test the FOSVS together with the AME in one of their UOG wells withdrew their offer. We have substituted the field test with SWN with a test in a Core Energy well in Michigan and with extensive testing and measurements in our laboratory.

Fluidion was never able to miniaturize their Acoustic Micro Emitters (AME) so we replaced the Fluidion AME with AME from Terves located in Euclid, Ohio.

End Budget Period IV – end of Project

Appendix 1. General Project Benefits and Dissemination of Project Information

What opportunities for training and professional development has the project provided?

Our Sr. Mechanical Engineer is training our machinists on high volume production. We purchase two large CNC lathes with milling capabilities. Our machine shop staff is trained on these machines. Our recently hired electro-optical engineer/scientist is working closely with our senior system engineers to first study and then further develop our optical testing and optical modeling techniques. Our optical staff has worked closely with our seismic staff to learn the essential seismic attributes that the optical seismic instruments must provide. Our seismic staff has worked with the optical staff and learned the limiting parameters of optical sensor technologies.

During the first quarter FY17 Paulsson interviewed potential employees from the Exceptional Children's Foundation. This is a program to train young adults on the autistic spectrum as machinists. We hired two your adults who started with Paulsson in January 2, 2017. The two new machinists have already contributed significantly to the manufacturing of our new Fiber Optic Seismic Vector Sensor (FOSVS) system.

How have the results been disseminated to communities of interest?

Dr. Björn Paulsson gave an invited presentation at the USEA workshop in Washington DC on December 18, 2014 with the title "**New Subsurface Signals are Needed for Improved Imaging of Subterranean Reservoirs & Resources**". This workshop series is sponsored by US DOE. The presentation was very well received. The presentation is available on www.usea.org.

Björn Paulsson gave a presentation at a RPSEA workshop in January 2015.

Bjorn Paulsson gave an oral presentation and published a paper at The World Geothermal Congress (WGC) in April 2015. The conference was held in Melbourne Australia.

Bjorn Paulsson attended the DOE Peer review meeting for Geothermal technology in May 2015 and presented a paper and report. Our project received an average score of 9.0 out of possible 10.

Our project was presented at the AAPG meeting held in Denver, CO in June 2015 at the RPSEA booth. Bill Head at RPSEA presented the project.

Dr. Paulsson presented the project in the DOE booth at the Offshore Technology Conference (OTC) held May 4 – 7, 2015 in Houston.

Dr. Paulsson presented the project to the RPSEA TAC committee meeting held June 6 – 9, 2015 in Houston.

Dr. Paulsson attended UrTEC meeting in San Antonio, TX in July 2015 and gave a poster presentation on our fiber optic technology.

Dr. Paulsson gave a presentation at the RPSEA meeting in September 2015.

Dr. Paulsson participated in the RPSEA booth at the SPE annular meeting in September 2015. The meeting was held in Houston.

Dr. Paulsson gave an invited Distinguished lecture presentation at USC in December 2015. Dr. Paulsson gave a presentation in June 2016 to an industrial TAC committee at RPSEA.

Dr. Björn Paulsson gave an invited luncheon presentation to 200 industry and government representatives at the RPSEA workshop "**Best of RPSEA 10 years of Research**" in Galveston, TX on August 30, 2016 with the title "**Fiber Optic Seismic Vector Sensor (FOSS) tracking of Acoustic Micro Emitters (AME) to Optimize Unconventional Oil and Gas (UOG) Development**" The presentation was very well received.

Paulsson, Inc. exhibited at the SEG annual convention held that was held in October 2016 in Dallas, TX. The products show cased included both the FOSS sensors and the AME's. Considerable interest was shown in both products. The Paulsson team also presented a paper at the post-convention workshop on optical sensor technologies.

Paulsson, Inc. exhibited at the SEG annual convention held that was held in October 2017 in Houston, TX. The products show cased included both the FOSVS sensors and the AME's. Considerable interest was shown in both products. The Paulsson team also presented a paper at the post-convention workshop on optical sensor technologies.

Paulsson presented a paper at the DOE Subsurface conference in Pittsburgh during August 2018.

Paulsson exhibited at the SEG conferences held in Anaheim, CA during August 14-17, 2018.

The Paulsson team will present a paper on the downhole seismic vibrator source at the Stanford Geothermal conference in Palo Alto, CA in February 2019.

Paulsson, Inc. is participating in conferences and workshops showcasing its optical sensor technologies. This FY we expect to participate in three conferences.

Paulsson presented a paper at the DOE Subsurface conference in Pittsburgh during August 2019.

Paulsson exhibited at the SEG conference held in San Antonio, TX September 15-18, 2019.

Appendis 2: PRODUCTS: What has the project produced?

Publications, conference papers, and presentations

Dr. Björn Paulsson gave an invited presentation at the USEA workshop in Washington DC on December 18, 2014 with the title "**New Subsurface Signals are Needed for Improved Imaging of Subterranean Reservoirs & Resources"**. This workshop series is sponsored US DOE. The presentation was very well received. The presentation is available on www.usea.org.

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Dr. Paulsson attended UrTEC meeting in San Antonio, TX in July 2015 and gave a poster presentation on our fiber optic technology funded by DOE.

Dr. Paulsson gave a presentation at the RPSEA meeting in September 2015.

Dr. Paulsson participated in the RPSEA booth at the SPE annual meeting in September 2015 and gave numerous presentations to conference attendees. The meeting was held in Houston.

Dr. Paulsson gave an invited Distinguished lecture presentation at USC in December

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Dr. Björn Paulsson gave an invited luncheon presentation to 200 industry and government representatives at the RPSEA workshop "**Best of RPSEA 10 years of Research**" in Galveston, TX on August 30, 2016 with the title "**Fiber Optic Seismic Vector Sensor (FOSS) tracking of Acoustic Micro Emitters (AME) to Optimize Unconventional Oil and Gas (UOG) Development**" The presentation was very well received.

The Paulsson team presented a paper at the SEG fiber optic seismic sensor workshop in Dallas, TX in October 2016.

The Paulsson team presented a paper and a poster at the IEAGHG CO2 conference in Traverse City in June 2017.

The Paulsson team presented a paper at the DOE unconventional conference in Pittsburgh, PA in August 2017.

The Paulsson team presented a paper at the SEG fiber optic seismic sensor workshop in Houston, TX in September 2017.

The Paulsson team presented a paper at the DOE subsurface conference in Pittsburgh, PA in August 2018.

The Paulsson team presented a paper on the downhole seismic vibrator source at the Stanford Geothermal conference in Palo Alto, CA in February 2019.

The Paulsson team will present a paper and a poster at the DOE subsurface conference in Pittsburgh, PA in August 2019.

The Paulsson team will present a paper at the World Geothermal Conference (WGC) held in Reykjavik, Iceland in May 2020.

Website(s) or other Internet site(s)

A discussion on our new Fiber Optic Seismic Vector Sensors (FOSVS) is now available on www.paulsson.com.

Technologies or techniques

The fiber optic sensor developed under DE-FE0024360 is being used for geotechnical applications.

Inventions, patent applications, and/or licenses

We received a patent on the drill pipe based deployment system for the optical sensors.

We have a patent application for our optical vector sensor in the process to be submitted to the patent office.

Other Products and Commercial Projects

Distributed Temperature Sensor (DTS) and Distributed Acoustic Sensors (DAS) can be combined with the Fiber Optic Seismic Vector Sensors (FOSVS).

NIOSH requested a proposal to use the FOSVS to monitor mines.

Several Oil Companies have requested a proposal to use the FOSVS to monitor oil and gas production.

A geotechnical company requested a proposal to use the FOSVS to monitor a geothermal development project.