

# Oil & Natural Gas Technology

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## Final Report

### Supercement for Annular Seal and Long-Term Integrity in Deep, Hot Wells “DeepTrek”

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Office of Fossil Energy

*DeepTrek Project Final Report  
October 2003 through August 2007*

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## *October 2003 through August 2007*

### **Abstract**

The purpose of this project is to formulate a “Supercement” designed for improving the long-term sealing integrity in HPHT wells. Phase I concentrated on chemistry studies and screening tests to design and evaluate Portland-based, hybrid Portland, and non-Portland-based cement systems suitable for further scale-up testing. Phase II work concentrated on additional lab and field testing to reduce the candidate materials list to two systems, as well as scaleup activities aimed at verifying performance at the field scale. Phase II was extended through a proposal to develop additional testing capabilities aimed at quantifying cementing material properties and performance that were previously not possible. Phase III focused on bringing the material(s) developed in previous Phases to commercialization, through Field Trials, Cost/Benefit Analysis, and Technology Transfer.

Extensive development and testing work throughout the project led to Phase III commercialization of two very different materials:

- Highly-expansive cement (Portland-based), patent pending as “PRESTRESSED CEMENT”.
- Epoxy Resin (non-Portland-based), patent pending. Trade name is Ultra Seal-R.

In Phase III, work concentrated on application of the Supercement materials in various increasingly-challenging wells. Previous testing revealed that PRESTRESSED CEMENT, when applied in weak or unconsolidated formations, tends to expand away from the central pipe, restricting the applicability of this material to competent formations. Tests were devised to quantify this effect so the material could be applied in appropriate wells. Additionally, the testing was needed because of industry resistance to expansive cements, due to previous marketing attempts with other materials that were less than successful. Field trials with the Epoxy Resin currently numbers in the hundreds of jobs at up to 295 deg F, with a large percentage being completely successful.

Both the PRESTRESSED CEMENT as well as the Ultra Seal-R represent materials fulfilling the objectives of the DeepTrek project.

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## **Introduction**

With current completion technology, oil and gas wells completed in high pressure and high temperature (HPHT) environments often experience escalating costs over the life of the well due to loss of sealant integrity. High temperatures are generally defined as those in excess of 350 deg F, and high pressures are those in excess of 15,000 psi. In this context, these high life-cycle costs are related to both the loss of production as a result of annular seal failure and the resulting remedial repair. Remedial procedures for restoring seal integrity in HPHT wells are significantly more expensive than in non-HPHT wells, and are often repeated during the life of the well in order to maintain annular seal. In extreme conditions, loss of annular seal can result in well abandonment and potential environmental and safety issues.

This project, as part of the Department of Energy (DOE) Deeptrek project, focuses on improving the economics associated with drilling deep, hot wells by developing new cementing materials engineered to provide long-term annular sealing in high-stress environments.

The project encompasses:

- Literature search
- Chemical cement design
- Lab screening testing
- Testing development
- Manufacturing and mixing scale-up
- Full-scale test applications and evaluations
- Technology transfer

This report details not only the third project phase (Commercialization), but recaps the results of the entire three-year program. Phase III work concentrated on performing Field Trials with the Supercement materials, Commercialization, and Technology Transfer.

## **Project Objectives – Entire Project**

The third phase of this project concentrated on additional laboratory test work, scaleup activities, and the development of new testing equipment and protocols. These activities served to reduce the number of candidate materials identified in Phase I to those with the best chance of meeting the project goals. At the end of Phase II, two materials remain for full field testing, economic evaluation, and commercialization in Phase III.

## **Phase I Summary**

### *Phase I Work Plan – Identification and Evaluation of Materials*

#### *Task 1: Perform a literature search of materials*

- Portland binders
- Non-Portland binders
- Admixtures for mechanical integrity

#### *Task 2: Evaluate the performance of selected materials at low temperature and identify top performers.*

- Conduct a preliminary laboratory screening to determine the effectiveness of binders under typical oilwell conditions.
- Screen selected admixtures for their ability to improve the mechanical properties of the supercement including mixability, strength/resilience, and durability.
- Perform laboratory tests to develop ways to control the set time and consistency in a field application with various chemicals such as retarders, accelerators, fluid-loss additives, and additives used for controlling gas migration.

#### *Task 3: Evaluate the performance of selected materials at high temperatures (>350°F) and pressures (15,000 psi) and identify top performers.*

Conduct laboratory tests to determine the effect of high temperatures and pressures on the following mechanical properties: thickening time, consistency, mixability, and set properties. Conduct preliminary mechanical testing of selected compositions using unconventional test methodologies such as tensile strength, Young's modulus, anelastic strain, and temperature and pressure cycling tests to determine which materials will be tested in Task 4.

#### *Task 4: Perform an in-depth evaluation of compositions using unconventional test methodologies.*

- Test selected compositions for anelastic strain, effects of temperature and pressure cycling, expansion, bonding, permeability, and annular seal.
- Perform numerical modeling of laboratory performance to aid in the prediction of stress/strain performance envelopes.

**Phase I—Major Findings and Conclusions**

1. Ceramicrete is not a viable material for use in deep, hot wells.
2. Ten (10) candidate formulations have been identified for further study. These systems represent:
  - Conventional Portland cement slurries (for baseline comparison only)
  - Portland cement with unconventional additives
  - Portland cement with unconventional amounts of conventional additives
  - Non-Portland formulations

Table 1 shows the specific formulations that were generated by Phase I work.

<i>System</i>	<i>Formula</i>	<i>Recipe</i>	<i>Water</i>	<i>Density</i>
Baseline	77	H+25% SBMC+15%MFA+2% Daxad 19	2.5	19.7
Baseline	99	H+35% Silica Flour	5.16	16.6
MgO	128	Baseline 77+20% MgO H	3.6	19.0
Moly	132	Baseline 99+0.5% Moly	5.19	16.6
Moly	133	Baseline 77+0.5% Moly	2.5	19.7
Resin	120	Yellow HT Resin+Activator	N/A	
Resin	121	Red HT Resin+Activator	N/A	
Fiber	130	Baseline 99+1.0% Ceramic Fibers	5.21	16.6
Fiber	131	Baseline 99+1.4% Ceramic Fibers	5.24	16.6
Fiber	136	Baseline 77+0.5% Ceramic Fibers	2.5	19.6
Ca Al Silicate	169	Na <sub>2</sub> SiO <sub>3</sub> + Calcined Lime + Calcined Alumina + Boehmite + Silica Flour	3.6	15.4

**Table 1 - Candidate System Formulations**

3. Although resin possesses some intriguing properties, mixing and handling difficulties may be significant in the field. Evaluation will continue into Phase II.
4. More refined high temperature testing protocols are required to determine and fully evaluate the ability of materials to provide an effective annular seal.
5. No single material property is sufficient to determine annular seal effectiveness.
6. Annular seal effectiveness is determined by an interaction between many mechanical properties. While some work has been done to quantify this relationship, it is not fully defined.



## **Phase II Summary**

### ***Phase II Work Plan – Manufacture and Scaleup Testing of Cement***

#### ***Task 1: Manufacture Supercement to specification***

- Manufacture the Supercement in a pilot plant to assess its performance on a larger scale
- Manufacture the Supercement in actual industrial quantities at a full-scale facility to finalize the manufacturing method that will be used to produce the cement commercially.

#### ***Task 2: Perform conventional and nonconventional batch testing to confirm the product's performance on a commercial scale.***

- Perform standard laboratory testing of the material manufactured in Task 1 to assess the effectiveness of the compositions for field applications.
- Tests used for this task will be the same as those used in the laboratory testing of selected compositions in Phase 1, Task 3.

#### ***Task 3: Evaluate the product's performance in large-scale mixing, shearing, and drillout studies.***

- Blend and mix field-application-size batches of the material with oilfield blending and mixing equipment.
- Perform laboratory tests to confirm the performance of the blended composition.
- Shear the material through a pipe loop to simulate placement in a well.
- Perform laboratory tests on the slurry to reconfirm performance after shearing.
- Create large-scale drilling targets to determine the rate of penetration with standard drilling equipment.
- Develop procedures for blending, mixing, placement, and drillout.

#### ***Task 4: Test the composition's performance in a research test well.***

Design a cement slurry as appropriate for the well conditions.

Blend, mix, and pump the slurry into the test well.

Conduct a post-job evaluation of the set slurry using logging results and pressure tests.

- Report on the effectiveness of binders at high temperatures and determine the best candidates for continued evaluation in Phase II.

#### ***Task 5: Develop Apparatus and Conduct tests for HPHT Annular Seal, Direct-pull tensile test, and HPHT Continuous Expansion.***

Through an approved project extension associated with Phase II, apparatus were developed to measure various performance and properties of cementing materials. Before this extension, these apparatus were not available.

- HPHT Annular Seal – take protocol developed at ambient pressure and temperature to evaluate the annular sealing performance of various cement materials at high temperature and pressure.

- HPHT Expansion – measure continuously the expansion or contraction of cement during cure and post-cure.
- Direct-pull Tensile Test – Economically measure the tensile strength of cement in a direct-pull method, in a compression test machine.

### *Phase II – Major Findings and Conclusions*

1. The original slate of 10 candidate sealants was reduced to two systems for Field Evaluation in Phase III. These systems are a non-shrinking Epoxy Resin and a highly-expansive Portland cement slurry design (“PRESTRESSED CEMENT”). Patents are pending on both products.
2. Both sealants are controllable through a wide range of temperature conditions, making them viable materials for wellbore sealants at high temperatures.
3. Epoxy Resin is believed to seal through a different mechanism than conventional cements, relying on mechanical means and the compliant nature of the Epoxy Resin material, rather than inherent matrix strength and chemical bonding. Multiple field trials have proven the ability of the material to seal in conditions under which conventional Portland cements had repeatedly failed.
4. PRESTRESSED CEMENT has exhibited significantly better performance in the lab than conventional Portland Cements, due to the highly-expansive nature of the material. When cured under confined conditions, the expansion creates an internal compressive preload that enables the material to better resist induced tensile stresses in the well than conventional cements. Additionally, the material exhibits very high mechanical shear bond and hydraulic bond.
5. Through the Phase II Extension phase, tests were developed to measure various performance and property characteristics. The tests have been successful to varying degrees:
  - Measure the annular sealing potential of various sealants under High Pressure and Temperature conditions.
  - Measure the expansion / contraction of cement continuously at High Pressure and Temperature conditions, during and after hydration.
  - Measure the tensile strength (and tensile fatigue characteristics and tensile Young’s Modulus, if desired) of sealants, using a direct pull method and conventional compressive test machine.

## **Phase III Project Work Plan**

### ***Phase III—Evaluate the Supercement in Field Applications***

#### ***Task 1—Evaluate the supercement in field applications.***

- Formalize plans with major operators to cement three to six wells in deep, hot conditions, with several different performance envelopes. Operators including Anadarko, EOG Resources, El Paso Natural Gas, Newfield Exploration Co., Conoco, and Chevron have ongoing working relationships with CSI and have expressed interest in collaborating with CSI in field-testing new products and processes to advance cementing technology. These operators have been working in South Texas, and shallow water GOM.
- Design the field-application job in accordance with well conditions.
- Complete laboratory testing for job design analysis.
- Schedule onsite consultants to ensure proper application of the cement system.
- Perform a post-job analysis of the cement system's performance as indicated by the evaluation of logs and pressure tests.

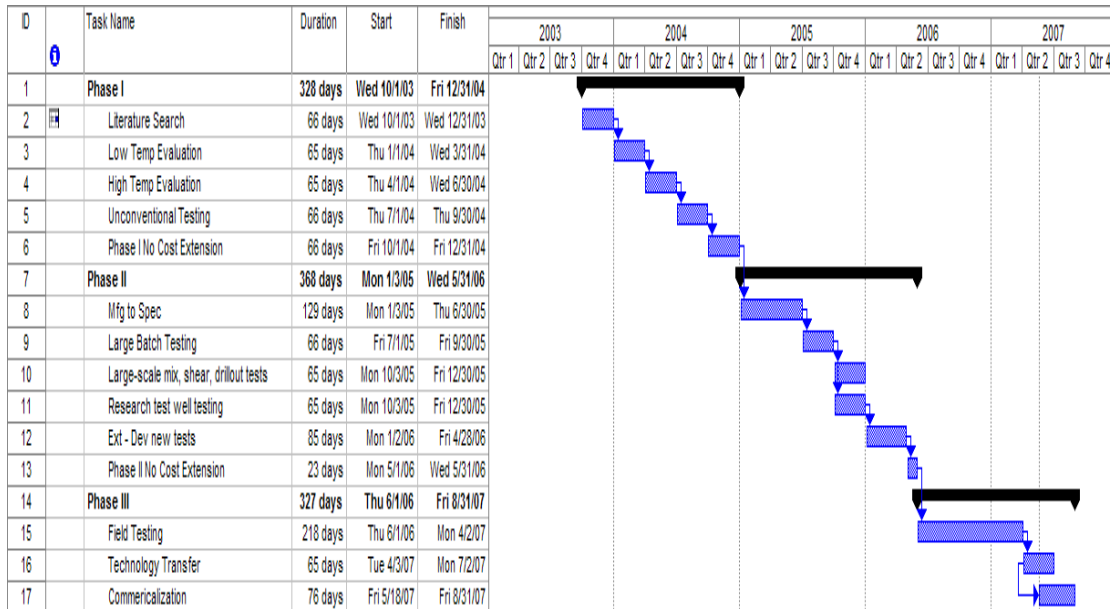
#### ***Task 2 —Perform a cost benefit analysis.***

- Analyze the technical benefits of using the supercement to provide zonal isolation in deep, hot wells.
- Analyze the economic benefits of the new supercement product to determine whether its manufacture is commercially feasible.

#### ***Task 3—Develop opportunities for technology transfer.***

- Organize workshops in Gulf Coast and West Texas region by working with the PTTC.
- Publish an article for the SPE/IADC conference on the benefits of the supercement and its performance in field applications.

## Project Schedule



## Phase III / Project Conclusions

1. Ultra Seal-R has proved to be a revolutionary new material with the capability to provide exceptional annular sealing performance in HPHT wells. Previous industry resin applications had drawbacks such as shrinking and intolerance to water contamination, whereas Ultra Seal does not suffer from these drawbacks. Ultra Seal Resin is also inert to chemical attack that typically degrades Portland cements over time. The material remains significantly more flexible than Portland cements, allowing it to move with stress rather than crack as do more brittle materials.
2. Ultra Seal-R believed to seal through a different mechanism than conventional cements, relying on mechanical means and the compliant nature of the Epoxy Resin material, rather than inherent matrix strength and chemical bonding. Multiple field trials have proven the ability of the material to seal in conditions under which conventional Portland cements had repeatedly failed.
3. PRESTRESSED CEMENT provides significantly higher resistance to tensile stresses developed in a cement sheath, by developing high in-situ compressive stresses during curing under confining conditions. This residual stress must be relieved before the material goes into tension, increasing the effective tensile strength of the material. Application is limited to wells that provide enough confinement to develop the internal stresses.

4. Economic Analysis shows that current Ultra Seal-R price is significantly more than conventional cements. The cost cannot be offset by savings in lost production and reduced need for remedial repair of annular seal over the life of the well if all sealants are replaced to Ultra Seal-R. However, the use of this material across critical points in the well subject to maximum stress conditions (e.g. as a substitute for tail cements on the casing strings) can lower the total cost of drilling and operating a well by reducing repair cost over the life of the well.
5. Economic Analysis shows that PRESTRESSED CEMENT application in wells with competent downhole formations and subject to significant pressure events during the life of the well can significantly reduce the need for remedial repairs at a cost comparable to conventional Portland cements.
6. Successful field trials were conducted with both materials, and Ultra Seal-R was awarded Hart's E&P 2007 Meritorious Award for Engineering Innovation.
7. Both materials were presented at the 2007 IADC/DEA Workshop, held at Moody Gardens, Galveston in June, 2007.

## **Phase III Results and Discussion**

### ***Task 1 – Evaluate the Supercement in Field Applications***

#### ***Ultra Seal Resin Field Jobs***

Ultra Seal-R has been used in over 50 field jobs, at temperatures up to 210 degrees F for pumped jobs and 295 degrees F for dump bailer applications. 92 percent of all jobs performed to date have been declared successful in providing a seal for the intended objectives. A great variety of jobs have been performed, including PTA, as well as primary, squeeze, liner, and a wide variety of plugging jobs.

Field work was begun in simple low-temperature PTA applications. There are several cases in which operators had spent millions of dollars with conventional cements to plug wells without success. Due to these failures as well as the unique properties of the material, a single application of Ultra Seal-R resulted in an effective plug, allowing the operator to abandon the well. Ultra Seal-R has many unique properties that are very different than conventional Portland cements, which opens up innovative placement techniques. Through the development process, new placement techniques were developed and tested at a rapid pace, as customers presented new challenging problems. Simultaneously, a resin database was designed to capture the various formulations of the material to enable the Application Engineers to rapidly choose recipes appropriate for the intended application. Formulation development was done at increasing temperatures with alternate constituents to be able to control the set. The unique properties presented by Ultra Seal-R include:

Ability to be lightened or weighted such that it can be lighter than, heavier than, or the same density as the wellbore fluid.

Ultra Seal-R does not mix with wellbore fluids, so heavy resin can be poured through standing fluids without intermixing.

Ultra Seal-R can be lightened so it floats on top of wellbore fluids, or formulated for neutral balance.

The native material contains no solids, so it can be injected deep into permeable formations without screening out due to particles that are too large to enter the formation. This makes it possible to create a “virtual wellbore”, and to consolidate unconsolidated formations. An example of an effective use of this property in a non-cement application includes refurbishing gravel packs.

A job reports for an unusual application is presented in the Appendix. This job involved pumping Ultra Seal-R down ¼” coiled tubing over 9,000 feet. Although there were issues with friction pressure because caused by the very small tubing and resultant heat generations issues, the job report indicates the job was completed and the results were successful. Additional data included in the Appendix includes several dump bailer designs up to 340 deg F. These jobs were not performed for various reasons unrelated to the material, but the laboratory results indicate that the material can be formulated and controlled at these temperatures. Work is continuing to increase the application temperature further with different extenders and hardeners.

As noted in the reports for both Phase I and Phase II, handling and mixing is significantly different than conventional oilfield cements. However, CSI personnel have developed a fit-for-purpose small volume mixing and pumping skid for these jobs. Readily-available personal protection equipment (PPE) is required for those handling or breathing fumes from the material, and the use of a methanol and water blend for cleanup presents a relatively simple disposal issue. Concepts have been developed for continuous mixing equipment, but job volumes to date have not made that equipment economically viable. While mixing procedures and equipment differ from conventional oilfield experience, relatively simple equipment and precautions make the use of Ultra-Seal R safe and effective.

### ***PRESTRESSED CEMENT Field Jobs***

Field applications of PRESTRESSED CEMENT cement have been somewhat more difficult than those involving Ultra Seal-R. Expanding cements are not new; some of the same materials used in PRESTRESSED CEMENT has been used for years to at least combat the natural volumetric shrinkage of Portland cement as it cures, and in some cases to generate a modest expansion. Marketing efforts by several major service companies focused on the advantages of the expansion, but the amount of expansion was not high enough to generate the internal prestress of PRESTRESSED CEMENT. Additionally, application in poorly-confined environments resulted in poor bond between the cement and internal pipe, adding to the disappointing performance in the field. The failure of these materials to live up to the marketing promises has led to general skepticism in the market regarding the benefits of the expansion. The key to success of PRESTRESSED CEMENT is not only the expansive characteristics, but also the microfine materials that are added to augment the performance. Success in the market is directly related to the discipline to not recommend the material in applications in which downhole confinement is not high enough to allow the material to function properly. With sufficient confinement, the material can achieve superior results, but it can also disintegrate during curing if the confinement is too low.

No field jobs have been performed to date with PRESTRESSED CEMENT. Two jobs were scheduled for Goldking Operating, but were cancelled for various reasons. Work is continuing to secure field jobs with the material. Although the Goldking jobs (a squeeze job and a tail-in slurry on a primary job) were not performed, all engineering and lab testing was completed in preparation. The complete laboratory data is included in the Appendix. In anticipation of actual field jobs a full scale mixing of the PRESTRESSED CEMENT was conducted at a yard facility. The complete report is included in the Appendix.

## ***Task 2 – Perform a Cost / Benefit Analysis***

### ***Ultra Seal Resin Technical Benefits***

Ultra Seal Resin is believed to achieve effective annular seal through somewhat different mechanisms than conventional Portland cements. The material is resistant to stresses inducing strain in the sealant material, and is chemically inert in the wellbore environment, including

those that can degrade Portland cements over time.

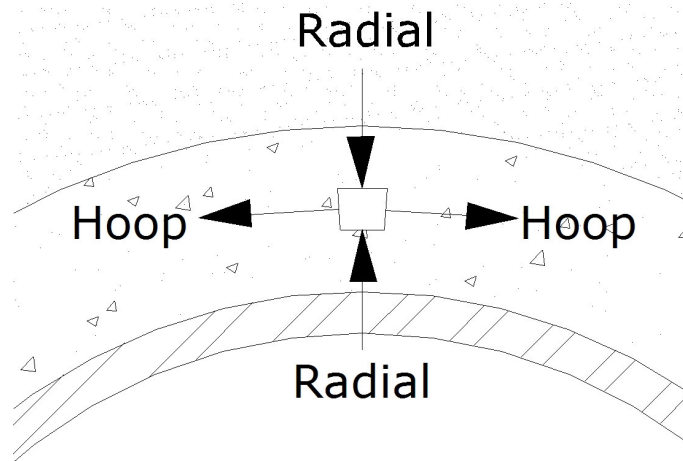
- Sealing performance appears to be a function of mechanical interference and chemical bond to the wellbore tubulars and formation. In an actual well, casing collars in the tubular string and discontinuities in the drilled wellbore present out-of-plane (with respect to the wellbore vertical axis) surfaces on which the material may impinge. The net effect is that the material behaves like an enormous pressure-activated elastomeric packing in the wellbore, sealing by mechanical interference means and chemical bonding.
- Laboratory evaluation of compressive and tensile strength was hampered by difficulty in judging when the material fails. With conventional Portland cements, which tend to be relatively brittle, failure occurs suddenly and obviously, usually accompanied by an audible crack and an immediate reduction in applied force. With Ultra Seal Resin, the material tends to compress and continue compressing as forces continue to increase without visible failure of the matrix. Further, removal of the applied force results in gradual growth of the sample back to nearly its original dimensions. This highly ductile behavior means that in a wellbore, applied stress to the resin sheath results in significant deformation without the creation of fluid-migrating cracks or fissures in the matrix.
- Conventional Portland cements are prone to long-term degradation due to exposure to many wellbore fluids, such as Carbon Dioxide. In wells that have these chemicals, initial sealing performance may be acceptable, but long-term integrity can suffer. Ultra Seal Resin is chemically inert to all known wellbore fluids, so long-term sealing integrity is not compromised.

#### ***PRESTRESSED CEMENT Technical Benefits***

PRESTRESSED CEMENT expanding cement functions by expanding against confinement during the hydration process. As the cement tries to expand against this confinement, the forces are directed internally, creating significant levels of internal compressive stresses within the matrix. Because cement is generally strong in compression and weak in tension, this high initial compressive stress “pre-stresses” the cement much like steel reinforcing rods do in construction concrete. When wellbore tubulars are pressurized, a triaxial stress state is produced within the cement matrix, consisting of a compressive radial component, a hoop (tangential) tensile component, and a shear axial component. By preloading in compression, the cement has a higher resistance to induced tensile hoop stresses than do conventional non-expanding cements.



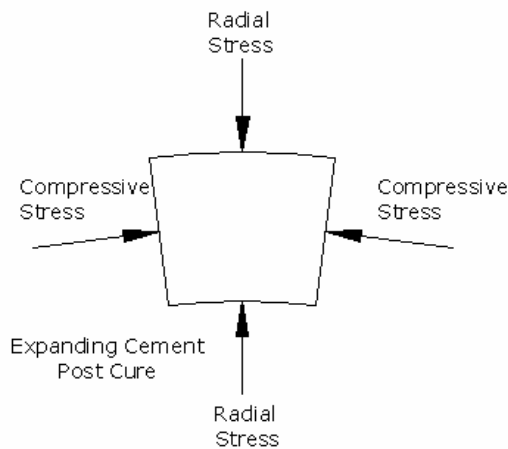
Figure 1 shows an infinitesimally small portion of cement and the stresses imposed upon it by internal pressure in the wellbore tubulars.



*Figure 1 – Stress State due to Internal Wellbore Pressure*

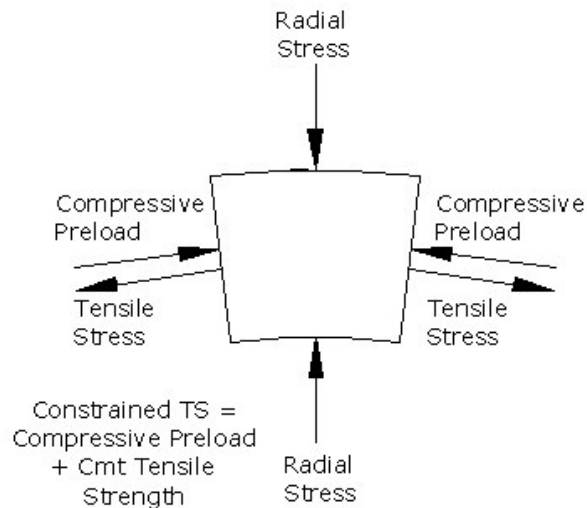
In the wellbore, if the induced tensile stress within the cement sheath becomes higher than the confined tensile strength of the material, a vertical crack will form in the material. This crack comprises a leakage path through which gas or other wellbore fluids can traverse the cement sheath.

PRESTRESSED CEMENT, expanding against confinement in the wellbore, creates an internal compressive preload as illustrated in Figure 2.



*Figure 2 – Initial Pre-Stressed State, Expanding Cement*

The compressive pre-load is generated as the cement tries to expand against the confinement provided by the external tubular, or the competent formation. When pressure is applied to the wellbore tubulars, the tensile stress generated in the cement sheath serves first to reduce the compressive pre-stress present in the cement before the material realizes a net tensile stress. Therefore, the effective compressive strength of the material is increased by the compressive preload applied (Figure 3).



*Figure 3 – Stress State in PRESTRESSED CEMENT under Load*

Any material without the in-situ compressive pre-load must bear the induced tensile stresses within the material matrix and the native tensile strength of the material. Although constrained tensile strength is higher than unconfined tensile strength, brittle Portland cement is inherently weak in tension and conventional cements fail more quickly due to imposed tensile stresses than do PRESTRESSED CEMENT in the wellbore environment.

The cost of PRESTRESSED CEMENT is comparable to conventional cements. Unlike Ultra-Seal R, in which higher initial costs are traded off for lower life cycle cost due to less requirement for remedial seal repair, there is less risk in using PRESTRESSED CEMENT. For wells in which the formations are competent, or for pipe-in-pipe applications, the substantially higher effective tensile strength means that the material will resist tensile stress-inducing intervention activities than will conventional cements.

#### ***Ultra Seal Resin and PRESTRESSED CEMENT Economic Analysis***

The overall economic benefit of improving annular seal performance in an HT-HP well is evaluated below. The typical well configuration, typical cements used as sealants, and required sealant volumes are presented in Figure 4 and Table 2. Table 3 lists typical well operations for a well such as the example. These operations and their typical frequency imply that a significant

number of stress cycles will be imposed on the annular sealants by hydraulic, thermal, and mechanical gradients.

Figure 4: Schematic of Wellbore used in Economic Analysis

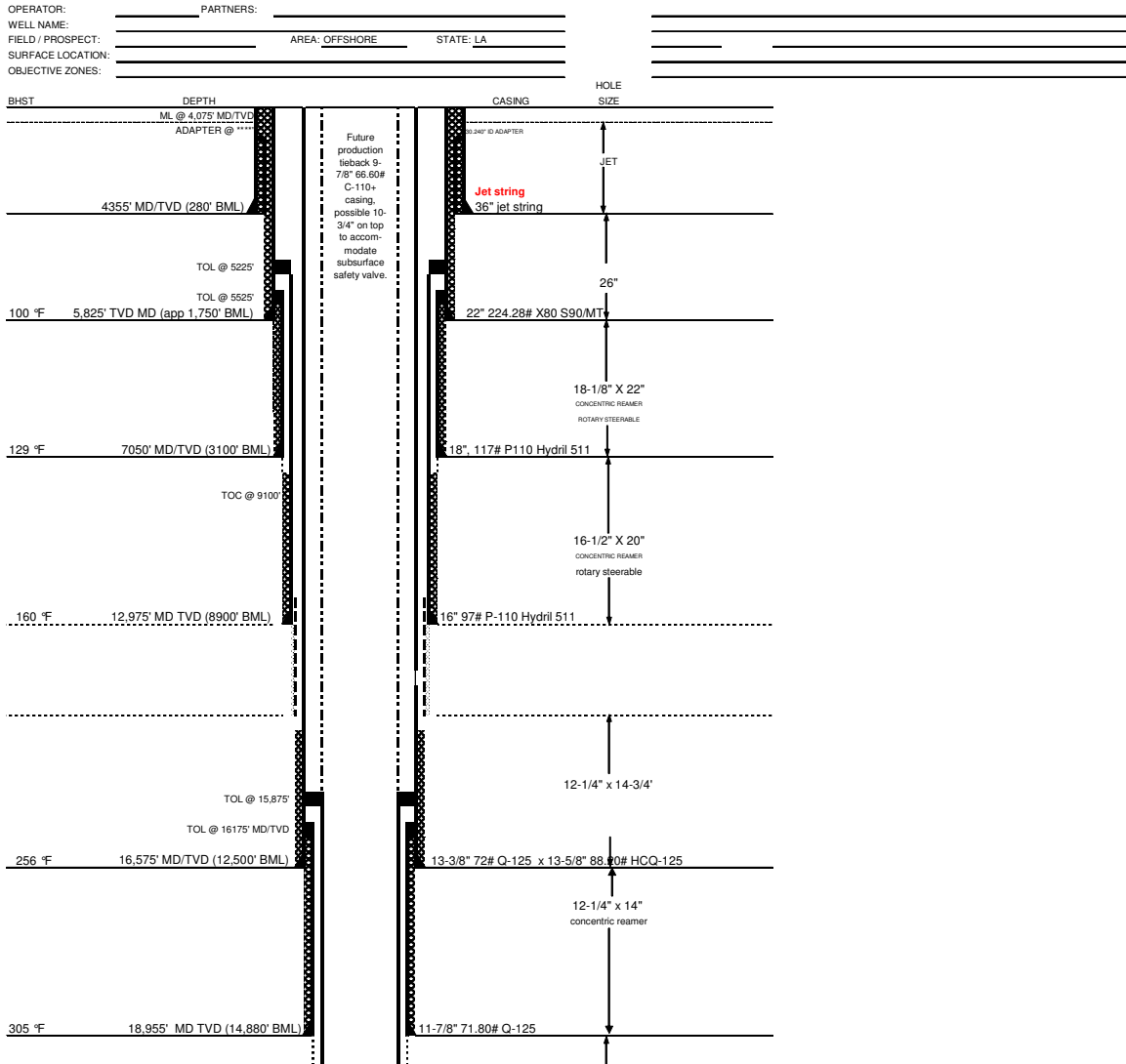


Table 2: Hole sizes, Depths, and Sealant Volumes

Pipe String	Hole Diameter (in)	Measured Depth (Top of Liner) (ft)	Top of Cement (ft)	Annular Sealant Volume (ft <sup>3</sup> )*	BHST (°F)
<b>Mud Line</b>		4,075			
<b>36-inch drive pipe</b>		4,355	na	na	na
<b>22-inch casing</b>	26	5,825	Mud line	3,150	100
<b>18-inch liner</b>	22	7,050 (5,525)			129
Lead			5,525	995	
Tail			6,550	485	
Total				1,480	
<b>16-inch liner</b>	20	12,975 (5,225)			160
Lead			9,100	3,180	
Tail			12,475	470	
Total				3,650	
<b>13-3/8-inch casing</b>	14-3/4	16,575			256
Lead			13,500	650	
Tail			15,575	130	
Total				780	
<b>11-7/8-inch liner</b>	14	18,955 (16,175)			305
Lead			16,175	2,000	
Tail			18,455	185	
Total				2,185	
<b>9-5/8-inch liner</b>	12 1/4	22,625 (19,125)			380
Lead			19,125	1,130	
Tail			22,125	185	
Total				1,315	

\*Volume of sealant required includes 20% excess in accordance with standard industry practice.

Table 3: Operations Inducing Stress on Annular Sealants

Operation	Number
Shoe and liner top tests prior to drill ahead	8
Replace drilling fluid with completion fluid	1
Perforate	2
Frac Pack	2
Acid treatment	1
Flow intervention	3/year
Plug to abandon	1

Assumptions made for this assessment regarding sealant composition and cost are presented in Table 4. Sealant compositions are generic, but they are based on sound cementing practices using materials that are available from all service companies. Prices reflect normal discounts currently given for cementing operations.

Table 4: Sealant Compositions and Cost

<b>Current Generic Cementing Compositions*</b>				
<b>Pipe String</b>	<b>Composition</b>	<b>Cost per ft<sup>3</sup> (\$)</b>	<b>Volume (ft<sup>3</sup>)**</b>	<b>Total Material Cost (\$)</b>
<b>36-inch drive pipe</b>	Not cemented			
<b>22-inch casing</b>	API Class H cement + 7% HS + 1% HP FLA + 3.6 lb/sk KCl + .2 gal/sk liquid CaCl <sub>2</sub> mixed with 8.7 gal/sk sea water Density = 13.5 lb/gal; Yield = 1.99 ft <sup>3</sup> /sk	\$55.06	3,150	<b>\$173,440.00</b>
<b>18-inch liner</b>				
Lead	API Class H Cement + 1.8 lb/sk KCl + .08 gal/sk FLA + 1 gal/sk Extender Density = 13.5 lb/gal; Yield = 1.74 ft <sup>3</sup> /sk	\$15.86	995	\$15,780.70
Tail	API Class H Cement + 1.8 lb/sk KCl + 0.06 HP FLA + 0.26 gal/sk CaCl <sub>2</sub> Density = 16.4 lb/gal; Yield = 1.11 ft <sup>3</sup> /sk	\$23.70	485	\$11,494.50
<b>Total</b>			<b>1,480</b>	<b>\$27,275.20</b>
<b>16-inch liner</b>				
Lead	API Class H Cement + 1.8 lb/sk KCl + 0.05 gal/sk FLA + 0.5 gal/sk Extender + 0.07 gal/sk Retarder Density = 15.0 lb/gal; Yield = 1.31 ft <sup>3</sup> /sk	\$16.07	3,180	\$51,102.60
Tail	API Class H Cement + 2.5 lb/sk KCl + 0.06 gal/sk HP FLA + 0.08 gal/sk Retarder Density = 16.4 lb/gal; Yield = 1.09ft <sup>3</sup> /sk	\$22.30	470	\$10,481.00
<b>Total</b>			<b>3,650</b>	<b>\$61,583.60</b>
<b>13-3/8-inch casing</b>				
Lead	API Class H Cement + 1.8 lb/sk KCl + 0.05 gal/sk FLA + 0.75 gal/sk Extender + 0.02 gal/sk HT Retarder Density = 14.5 lb/gal; Yield = 1.41 ft <sup>3</sup> /sk	\$14.90	650	\$9,685.00

Tail	API Class H Cement + 35% Coarse Silica + 1.8 lb/sk KCl + 0.1 gal/sk HP FLA + 0.03 gal/sk HT Retarder	\$26.30	130	\$3,419.00
	Density = 17.3 lb/gal; Yield = 1.26 ft <sup>3</sup> /sk			
Total			780	<b>\$13,104.00</b>
<b>11-7/8-inch liner</b>				
Lead	API Class H Cement + 35% Fine Silica + 1.8 lb/sk KCl + 0.05 gal/sk FLA + 0.5 gal/sk Extender + 0.02 gal/sk HT Retarder	\$15.66	2,000	\$31,320.00
	Density = 14.5 lb/gal; Yield = 1.81 ft <sup>3</sup> /sk			
Tail	API Class H Cement + 35% Coarse Silica 1.8 lb/sk KCl + 0.1 gal/sk HP FLA + 0.05 gal/sk HT Retarder	\$26.77	185	\$4,952.50
	Density = 17.3 lb/gal; Yield = 1.26 ft <sup>3</sup> /sk			
Total			2,185	<b>\$36,272.50</b>
<b>9-5/8-inch liner</b>				
Lead	API Class H Cement + 35% Fine Silica + 1.8 lb/sk KCl + 0.05 gal/sk FLA + 0.5 gal/sk Extender + 0.06 gal/sk HT Retarder	\$16.32	1,130	\$18,441.60
	Density = 14.5 lb/gal; Yield = 1.81 ft <sup>3</sup> /sk			
Tail	API Class H Cement + 35% Coarse Silica 1.8 lb/sk KCl + 0.1 gal/sk HP FLA + 0.08 gal/sk HT Retarder	\$27.48	185	\$5,083.80
	Density = 17.3 lb/gal; Yield = 1.26 ft <sup>3</sup> /sk			
Total			1,385	<b>\$23,525.40</b>
<b>Total all materials</b>				<b>\$332,109.80</b>
*Cements for the 22-inch casing and 18-inch liner mixed with sea water. All others mixed with fresh water.				
**Volume includes 20% excess over calculated annular volume.				
Cost of material is based on averages of current service company price books with application of average discounts.				
HS = low density, hollow spheres				
HP FLA = high performance fluid loss additive				
FLA = fluid loss additive				
HT Retarder = High Temperature				

A summary of operator's estimated average cost for repairing sealant failure including rate of occurrence of failure for each sealant application and cost of repairing a seal failure are summarized in Table 5. These estimated costs are conservative in that they assume only cost for a perfectly executed and successful squeeze. No extra expense of extended time or failure are built in.

This analysis yields an average repair cost incurred for failed sealant repair throughout the life of an average well is \$2,434,250. This value is obtained by multiplying the cost of each failure times the failure rate.

Table 5: Failure Rate and Cost of Repair

Failure Rate and Cost of Repair					
Estimates for rates of seal failure for each cemented string in the example are listed.					
Pipe String	Failure Rate*	Material and Service Cost (\$)*	Rig Cost (\$)*	Cost per Failure (\$)	Cost per Well Drilled (\$)
22-inch casing	not measured	Na	na	na	na
18-inch liner	25%	\$30,000	\$1,000,000	\$1,030,000	\$257,500
16-inch liner	25%	\$30,000	\$1,000,000	\$1,030,000	\$257,500
13-3/8-inch casing	25%	\$35,000	\$1,500,000	\$1,535,000	\$383,750
11-7/8-inch liner	80%	\$35,000	\$1,500,000	\$1,535,000	\$1,228,000
9-5/8-inch liner	15%	\$50,000	\$2,000,000	\$2,050,000	\$307,500
<b>Average total cost of failure per well</b>					<b>\$2,434,250</b>
*Estimates are based on informal survey of current HTHP well drillers and operators					

Price comparisons for standard cements and newly-developed cements appear in Table 6. Assumptions on which these price estimates are based are detailed in footnotes beneath each table. Data presented in Table 6 are derived assuming application of Pre-Stressed Cement similar to the composition mixed in the previously-discussed mixing trial. Prices for Pre-Stressed Cement were estimated starting with compositions similar to the standard ones listed in Table 4 and adding 30% MgO and 25% Microfine cement. Water, extender and retarder concentrations were adjusted to improve mixability and sufficient placement time.

Service company prices for MgO and Microfine Cement were estimated as five times material cost. This markup is not unusual for high-performance additives. No discounts were applied to these price estimates. Ultra Seal R price used is the undiscounted commercial price currently charged for the product: \$10,000/bbl.

Table 6: Price Comparison of Standard vs. Newly-developed Cementing Systems

	Yield (ft <sup>3</sup> /sk)				Price (\$/ft <sup>3</sup> )		
	Density	Current	Pre-Stressed	Ultra Seal*	Current	Pre-Stressed	Ultra Seal
<b>18-inch liner</b>							
Lead	13.5	1.74	2.59	5.61	\$15.86	\$64.48	\$1,782.50
Tail	16.4	1.11	2.02	5.61	\$23.70	\$79.35	\$1,782.50
<b>16-inch liner</b>							
Lead	15	1.31	2.00	5.61	\$16.07	\$77.38	\$1,782.50
Tail	16.4	1.09	2.02	5.61	\$22.30	\$79.36	\$1,782.50
<b>13-3/8-inch casing</b>							
Lead	14.5	1.41	2.17	5.61	\$14.90	\$71.48	\$1,782.50
Tail	17.3	1.26	2.21	5.61	\$26.30	\$77.71	\$1,782.50
<b>11-7/8-inch liner</b>							
Lead	14.5	1.81	2.61	5.61	\$15.66	\$62.85	\$1,782.50
Tail	17.3	1.26	2.21	5.61	\$26.77	\$78.85	\$1,782.50
<b>9-5/8-inch liner</b>							
Lead	14.5	1.81	2.61	5.61	\$16.32	\$64.20	\$1,782.50
Tail	17.3	1.26	2.21	5.61	\$27.48	\$80.62	\$1,782.50
*Ultraseal yield is expressed in ft <sup>3</sup> /bbl material to be consistent with current commercial pricing practice.							

The data in Table 6 reveals that the cost of Pre-Stressed cement is roughly 5 times greater than currently used cements. However, the current retail price of Ultra Seal R is around 80 times greater than those currently used. With an average maximum potential savings of \$2,434,250/well available if all the repairs are eliminated, Ultra Seal R can not compete based on price. Therefore, cost comparisons of Pre-Stressed cement vs. currently used compositions were calculated and appear in Table 7. The 20-inch casing string was not included in this comparison since these cements are specifically designed to stop shallow water flow. Direct substitution of Pre-Stressed cement components into this formulation would alter this cement's water-flow-stopping performance.

Table 7 indicates an overall cost increase of just over \$500,000 for the example well cemented with Pre-Stressed cement. Assuming that this action is successful in stopping only half of the current failures yields a remedial cost decrease of over \$1,000,000. This indicates the substitution is economically feasible.



Table 7: Cost Increase substituting Pre-Stressed Cement.

Pipe String	Current Material Cost (\$)	Pre-Stressed Material Cost (\$)	Increase (\$)
<b>18-inch liner</b>			
Lead	\$15,780.70	\$63,859.10	
Tail	\$11,494.50	\$38,484.75	
Total	<b>\$27,275.20</b>	<b>\$102,343.85</b>	<b>\$75,068.65</b>
<b>16-inch liner</b>			
Lead	\$51,102.60	\$246,068.40	
Tail	\$10,481.00	\$37,299.20	
Total	<b>\$61,583.60</b>	<b>\$283,367.60</b>	<b>\$221,784.00</b>
<b>13-3/8-inch casing</b>			
Lead	\$9,685.00	\$46,462.00	
Tail	\$3,419.00	\$10,102.30	
Total	<b>\$13,104.00</b>	<b>\$56,564.30</b>	<b>\$43,460.30</b>
<b>11-7/8-inch liner</b>			
Lead	\$31,320.00	\$125,700.00	
Tail	\$4,952.50	\$14,587.25	
Total	<b>\$36,272.50</b>	<b>\$140,287.25</b>	<b>\$104,014.75</b>
<b>9-5/8-inch liner</b>			
Lead	\$18,441.60	\$72,546.00	
Tail	\$5,083.80	\$14,914.70	
Total	<b>\$23,525.40</b>	<b>\$87,460.70</b>	<b>\$63,935.30</b>
<b>Total All Materials</b>	<b>\$161,760.70</b>	<b>\$670,023.70</b>	
<b>Cost increase using Self Stress Cement</b>			<b>\$508,263.00</b>

An HTHP well completed in a deep water environment was chosen as a high-profile example. Price and time estimates for well operations are averages of estimates from industry standard consensus. For comparison of cost of operations offshore and onshore, general operating costs for drilling or workover on land are assumed to be roughly 5 times less than those offshore. Therefore, substitution of Pre-Stressed Cement might not be economically feasible for onshore wells. However, most failures occur in the tail cement which represents a small portion of the overall sealant volume. Substituting Pre-Stressed Cement for the tail cement in the example represents approximately \$90,000 completion cost for the example well. This makes the substitution economically viable assuming a 50% failure reduction.

### *Task 3 – Develop Opportunities for Technology Transfer*

#### *Ultra Seal-R Meritorious Award for Engineering Innovation*

Ultra Seal-R was selected as one of the 2007 Hart E&P Meritorious Award for Engineering Innovation in the Drilling category. From the entries from across the Oil and Gas Industry, winners were chosen by an expert panel chosen from leading engineers and engineering managers from operating and consulting companies worldwide. The primary criteria for selection includes technologies that new and better methods for the increasingly-difficult task of finding and producing hydrocarbons. The announcement can be found at the following link: <http://eandp.info/area/meawinners> . The complete abstract submitted for the contest is included in the appendix, as well as the field jobs submitted for evidence that the technology is viable.

#### *2007 IADC / DEA Workshop*

The DeepTrek project was presented in the Completions for Deep/HPHT Wells II session at the 2007 IADC/DEA Workshop. This workshop was held at Moody Gardens in Galveston , June 19 & 20, 2007. The CSI presentation was conducted at 11:00 am on Wednesday, June 20. The presentation, entitled “Super-Cement for Annular Seal and Long-Term Integrity in Deep, Hot Wells was given by Mr. Fred Sabins, CSI president. The full content of the presentation as well as a synopsis of the Q&A following the presentation is included in the Appendix.

## **Appendices**

## *Appendix A – DeepTrek Phase I Data*

Phase I testing concentrated on three discrete tasks. The first was to evaluate candidate compositions at low temperatures. The second was to take the best candidate materials identified in the low-temperature screening and to evaluate them at high temperatures and pressures. The final testing task was to evaluate candidate materials with unconventional test methods, such as anelastic strain, expansion, annular seal, and other tests.

A total of 188 systems were evaluated during Phase I. Screening work was based primarily on basic mechanical properties, such as tensile and compressive strength. Some systems were not mixable and were quickly abandoned and no data was generated. The 188 individual systems (or specific recipes) were drawn from a number of different specific approaches to achieving improved properties. These approaches are collected in 21 categories, and are elaborated as follows:

- Baseline – these systems were evaluated as baseline, and represent typical solutions applied in HPHT wells today.
- Extended Portland cements, utilizing SMS and Bentonite
- Microflyash enhanced
- Microflyash / Microcement enhanced
- Microsilica enhanced
- Microflyash / Portland-based microcement enhanced
- Fiber enhanced, inert and reactive
- Miscellaneous systems
- Ceramicrete (non-Portland)
- Aluminum Phosphate (non-Portland)
- Hybrid Ceramicrete
- Coarse sand enhanced
- Reduced water
- High fluid-loss
- Miscellaneous non-Portland
- High Magnesium Oxide
- Calcium Phosphate (non-Portland)
- Zinc Phosphate (non-Portland)
- Ceramicrete / Aluminum Phosphate (non-Portland)
- Epoxy Resin (non-Portland)
- Molybdenum

Table A1 shows the complete results of Phase I testing. Reported are Low- and High-temperature compressive strength, compressive Young's Modulus, tensile strength, flexural strength, shear bond, annular seal, and anelastic strain. Note that not all fields are completed for every system; some were not mixable and some were evaluated and dropped from further consideration.



19	5.48		15% microflyash, 35% slag-based microcement		High	5,824	1,431,482	619	274									
20	4.36		15% microflyash, 35% slag-based microcement	17.9	Not Mixable													
21	3.48		15% microflyash, 35% slag-based microcement	18.8	Not Mixable													
22	5.48	Microsilicate	15% microsilica	15.7	Medium	2,869	442,452	354	242									
23	4.36		15% microsilica		High	2,975	388,589	788	286									
24	3.48		15% microsilica	17.6	Not Mixable													
25	5.48		25% microsilica		High	3,250	349,002	417	260									
26	4.36		25% microsilica		Not Mixable													
27	3.48		25% microsilica		Not Mixable													
28	5.48		35% microsilica		High	3,765	702,067	418	-									
29	4.36		35% microsilica		Not Mixable													
30	3.48		35% microsilica		Not Mixable													
31	5.48		Microflyash / Portland-based Microcement	15% microflyash, 15% portland-based microcement		Low	2,703	469,613	662	264								
32	4.36	15% microflyash, 15% portland-based microcement		17.3	Medium	3,462	749,747	473	526									
33	3.48	15% microflyash, 15% portland-based microcement		18.2	Medium	6,505	1,452,093	786	482									
34	5.48	15% microflyash, 25% portland-based microcement		16.6	Medium	4,734	659,972	589	305									
35	4.36	15% microflyash, 25% portland-based microcement		17.6	Medium	4,431	627,365	777	430									
36	3.48	15% microflyash, 25% portland-based microcement		18.5	High	5,897	795,351	932	-									
37	5.48	15% microflyash, 35% portland-based microcement		16.9	Medium	2,669	484,719	409	244									
38	4.36	15% microflyash, 35% portland-based microcement		17.9	High	7,217	1,228,805	737	619									
39	3.48	15% microflyash, 35% portland-based microcement		18.8	Not Mixable													

40	5.48	Fibers with Various Systems	2% SMS, 3.5% 1mm GF																	
41	5.48		15% microflyash, 3.5% 1mm GF																	
42	3.48		15% microflyash, 15% slag-based microcement, 3.5% 1mm GF																	
43	4.36		15% microsilica, 3.5% 1mm GF																	
44	5.48		25% microsilica, 3.5% 1mm GF																	
45	5.48		35% microsilica, 3.5% 1mm GF																	
46	5.48		15% microflyash, 15% portland-based microcement, 3.5% 1mm GF																	
47	5.48		2% SMS, 3.5% 1mm GF, 0.1 gps surf																	
48	5.48		15% microflyash, 3.5% 1mm GF, 0.1 gps surf	15.8																
49	5.48		15% microflyash, 15% portland-based microcement, 3.5% 1mm GF, 0.1 gps surf																	
50	5.48		2% SMS, 3.5% 6mm GF, 0.1 gps surf																	
51	5.48		15% microflyash, 3.5% 6mm GF, 0.1 gps surf																	
52	5.48		15% microflyash, 15% portland-based microcement, 3.5% 6mm GF, 0.1 gps surf																	
53	5.48		2% SMS, 3.5% 12mm GF, 0.1 gps surf																	
54	5.48		15% microflyash, 3.5% 12mm GF, 0.1 gps surf																	
55	5.48	15% microflyash, 15% portland-based microcement, 3.5% 12mm GF, 0.1 gps surf																		

56	3.48	Misc	35:65 Flyash:H			-	-	252											
57	3.48		35:65 Zeolite:H			-	-	343											
58	3.48		H + 15% Zeolite		Not Mixable														
59a		Ceramicrete	Ceramicrete, Class F, Dry, RT, 10 min, 48 hr			652													
59b			Ceramicrete, Class F, Dry, RT, 10 min, 1 wk			949													
59c			Ceramicrete, Class F, Wet, RT, 10 min, 48 hr			615													
59d			Ceramicrete, Class F, Wet, RT, 10 min, 1 wk			631													
59e			Ceramicrete, Class F, Dry, 350, 20 min, 24 hr																
59f			Ceramicrete, Class F, Wet, 350, 20 min, 24 hr																
60a			Ceramicrete, Class F, Dry, RT, 20 min, 1 wk			935													
60b			Ceramicrete, Class F, Wet, RT, 20 min, 1 wk			781													
61			Al Phos	Aluminum Phosphaste Cement, cured at high temp															
62		Aluminum Phosphaste Cement, cured at high temp																	
63		Hybrid Ceramicrete	10:90 Ceramicrete:H, 140 deg cure, 48 hr			1,800	137,965	392											
63A						1,350													
64			25:75 Ceramicrete:H, 140 deg cure, 48 hr			99	-	533											
64A						665													
65			50:50 Ceramicrete:H, 140 deg cure, 48 hr			552	67,355	410											
65A						538													
66			75:25 Ceramicrete:H, 140 deg cure, 48 hr			248	-	315											
66A						511													
67			90:10 Ceramicrete:H, 140 deg cure, 48 hr			819	106,491	315											
67A						163													



69		Coarse Sand	35% 100 mesh silica sand			-	-	418										
70			60% 100 mesh silica sand			-	-	478										
71			84.5% 100 mesh silica sand				-	-	431									
72			35% 20/40 mesh silica sand				-	-	472									
73			60% 100 mesh silica sand				-	-	398									
74			84.5% 100 mesh silica sand				-	-	489									
75	2.50	Reduced Water	35% microflyash, 2% daxad-19					763										
76	2.50		15% microflyash, 15% slag-based microcement, 2% daxad-19						968									
77	2.50		15% microflyash, 25% slag-based microcement, 2% daxad-19	19.7					1,237					471	10.3	3.49E-06		
78	2.70		15% microflyash, 15% portland-based microcement, 2% daxad-19			5,899			897									
79	2.50		15% microflyash, 25% portland-based microcement, 2% daxad-19		Not Mixable													
80	2.50		15% microflyash, 35% portland-based microcement, 2% daxad-19		Not Mixable													
81		High FL	Latex Liner System, 16.4 lb/gal			2,915	214,367	392										
82			Fluid Loss system w/ silica sand, 17.5 lb/gal			2,674	186,108	534										
83			Fluid Loss system w/ silica sand & hematite, 18.5 lb/gal			1,303	135,083	418										
84			Fluid Loss system w/ 18% KCl/NaCl mix, BWOW			1,118	90,944	368										

85		Non-Portland	10% alumina metaphosphate in cement		Cylinders	1,886		575		1,342	-	366					
85A					Cubes	2,204											
86			20% alumina metaphosphate in cement		Cylinders	1,494		465									
86A					Cubes	2,533											
87			10% sodium hexametaphosphate in cement														
87A						611											
88			20% sodium hexametaphosphate in cement														
88A						968											
89			10% bohemite in cement		Cylinders	1,385		388		751	-	357					
89A					Cubes	1,383											
90			20% bohemite in cement		Cylinders	1,558		426		1,243	-	377					
90A					Cubes	1,261											
91			1% sodium hexametaphosphate in cement		Cylinders	2,439		427		1,632	-	409					
91A					Cubes	2,341											
68		Mag Ox	20% MgO in Class H			2,990	422,499	318									
68A						2,092											
92	3.48		5% MgO L in cement	17.7		3577		733		1726		387					
93	3.48		10% MgO L in cement	17.9		6887		620		1155		393					
94	3.48		20% MgO L in cement	18.4		1351		639		459							
95	3.48		5% MgO M in cement	17.7		2172		614		1471							
96	3.48		10% MgO M in cement	17.9		2273		438									
97	3.48		20% MgO M in cement	18.4		1115		297									
98		HT Base	Class H, 16.4 lb/gal		350 deg / 3K psi, 1 day				2,765		357						
99			Class H + 35% silica flour	16.6	350 deg / 3K psi, 1 day				4,785		705		256	2.2	5.87E-06		

100		Calcium Phosphate	5% Calcium Phosphate in cement		350 deg / 3K psi, 1 day					1,411		289						
101			10% Calcium Phosphate in cement		350 deg / 3K psi, 1 day					1,394		251						
102			20% Calcium Phosphate in cement		350 deg / 3K psi, 1 day					960		274						
103		Zinc Phosphate	5% zinc phosphate in cement		350 deg / 3K psi, 1 day					1,329		250						
104			10% zinc phosphate in cement		350 deg / 3K psi, 1 day, Too soft													
105			20% zinc phosphate in cement		350 deg / 3K psi, 1 day, Too soft													
106		Fibers	Alumina with 50% phosphoric acid and aluminum hydroxide		350 deg / 3K psi, 2 day													
107			Class H + 35% silica flour + 3.5% Alumina Fibers	16.6	350 deg / 3K psi, 1 day					3503		625						
107			Class H + 35% silica flour + 3.5% 2mm Alumina Fibers		350 deg / 3K psi, 1 day					5649		725						
108			Class H + 35% silica flour + 3.5% 2mm Nylon Fibers		350 deg / 3K psi, 1 day					3229		670						
109			Class H + 35% silica flour + 3.5% 3mm Nylon Fibers		350 deg / 3K psi, 1 day					3018		753						
110			Class H + 35% silica flour + 3.5% 3mm Rayon Fibers		350 deg / 3K psi, 1 day					3412		439						
111		Ceramic / Al Phosphate	Ceramicrete		350 deg / atm, 1 day, dry, Catastrophic Expansion													
112			Ceramicrete		350 deg / 3K psi, 1 day, wet					190								
113			Ceramicrete		70 deg / atm, 1 day, dry	569												
114			Ceramicrete		100 deg / atm, 1 day, wet	1046												
115			Alumina Phosphate		350 deg / 3K psi, 72 hrs, wet													
116			Ceramicrete		70 deg / atm, 72 hr, dry (open lid)	882												

117		MgO w/ Sys 99	Class H + 35% silica flour + 5% MgO L		350 deg / 3K psi, 1 day, wet					3306	768				
118			Class H + 35% silica flour + 10% MgO L		350 deg / 3K psi, 1 day, wet					2660					
119			Class H + 35% silica flour + 20% MgO L		350 deg / 3K psi, 1 day, wet					3273					
120		Re sin	Yellow		230 deg / atm						2,206				
121			Red		230 deg / atm						3,199				
122	2.50	MgO / Sys 77	H+25% SB Microcement+15% Microflyash+2% Daxad 19+5% MgO L	19.8	350 deg / 3K psi, 1 day, wet						748				
123	2.50		H+25% SB Microcement+15% Microflyash+2% Daxad 19+10% MgO L	20.0	Not Mixable										
123A	3.28		H+25% SB Microcement+15% Microflyash+2% Daxad 19+10% MgO L	19.0											
124	2.50		H+25% SB Microcement+15% Microflyash+2% Daxad 19+20% MgO L	20.4	Not Mixable										
124A	3.60		H+25% SB Microcement+15% Microflyash+2% Daxad 19+20% MgO L	19.0											
125	5.22	Moly / Sys 99	Class H + 35% silica flour + 1% Molybdenum	16.6	350 deg / 3K psi, 1 day, wet					2,599	817				
126	5.32		Class H + 35% silica flour + 2.5% Molybdenum	16.6	350 deg / 3K psi, 1 day, wet					3,109	707				
127A	5.49		Class H + 35% silica flour + 5% Molybdenum	16.6	350 deg / 3K psi, 1 day, wet							532			
127B	5.49		Class H + 35% silica flour + 5% Molybdenum	15.4	350 deg / 3K psi, 1 day, wet							487			

128	3.60	Moly / Sys 77	H+25% SB Microcement+15% Microflyash+2% Daxad 19+20% MgO L	19.0	350 deg / 3K psi, 1 day, wet, Confined cure and cored sample tested						3,186		281		1850+		
129	3.28		H+25% SB Microcement+15% Microflyash+2% Daxad 19+10% MgO L	19.0	350 deg / 3K psi, 1 day, wet												
130	5.21	Fiber	H+35% silica flour+1.0% Ceramic Fiber	16.6	350 deg / 3K psi, 1 day, wet						3,709		1,016				4.04E- 06
131	5.24		H+35% silica flour+1.4% Ceramic Fiber	16.6	350 deg / 3K psi, 1 day, wet						3,021		1,149		186	50	3.10E- 06
132	5.19	Molybdenum	Class H + 35% silica flour + 0.5% Molybdenum	16.6	350 deg / 3K psi, 1 day, wet						4,383		1,083		2.3	1.55E- 06	
133	2.50		H+25% SB Microcement+15% Microflyash+2% Daxad 19+0.5% Molybdenum	19.7	350 deg / 3K psi, 1 day, wet						6,678		1,366				8.49E- 07
134	2.50		H+25% SB Microcement+15% Microflyash+2% Daxad 19+1.0% Molybdenum	19.7	350 deg / 3K psi, 1 day, wet						#####		1,137				
135	2.50		H+25% SB Microcement+15% Microflyash+2% Daxad 19+2.5% Molybdenum	19.8	Not Mixable												
136	2.50	Ceramic Fibers	H+25% SB Microcement+15% Microflyash+2% Daxad 19+0.5% Ceramic Fiber	19.6	350 deg / 3K psi, 1 day, wet								1,312				1.59E- 06
137	2.50		H+25% SB Microcement+15% Microflyash+2% Daxad 19+1.0% Molybdenum	19.7	350 deg / 3K psi, 1 day, wet								1,241				
138	2.50		H+25% SB Microcement+15% Microflyash+2% Daxad 19+1.5% Molybdenum	19.7	350 deg / 3K psi, 1 day, wet								1,280				

139	3.60	Sys 128-129 Restrained / Unrestrained	H+25% SB Microcement+15% Microflyash+2% Daxad 19+20% MgO L Restrained	19.0	350 deg / 3K psi, 1 day, wet														
140	3.60		H+25% SB Microcement+15% Microflyash+2% Daxad 19+20% MgO L Unrestrained	19.0	350 deg / 3K psi, 1 day, wet														
141	3.28		H+25% SB Microcement+15% Microflyash+2% Daxad 19+10% MgO L Restrained	19.0	350 deg / 3K psi, 1 day, wet														
142	3.28		H+25% SB Microcement+15% Microflyash+2% Daxad 19+10% MgO L Unrestrained	19.0	350 deg / 3K psi, 1 day, wet														
143	3.60	Fiber + Moly	H+25% SB Microcement+15% Microflyash+2% Daxad 19+20% MgO L	19.0	350 deg / 3K psi, 1 day, wet														
144	3.28		H+25% SB Microcement+15% Microflyash+2% Daxad 19+10% MgO L	19.0	350 deg / 3K psi, 1 day, wet														
145	5.25	HPHT Base w/ Reactive Fibers	H+35% silica flour+1.0% Ceramic Fiber+0.5% Moly	16.6	350 deg / 3K psi, 1 day, wet					5,456		1,007							
146	5.22		Class H + 35% silica flour + 0.5% Molybdenum+0.5% Ceramic Fibers	16.6	350 deg / 3K psi, 1 day, wet					6,782		1,043							
147	2.50		H+25% SB Microcement+15% Microflyash+2% Daxad 19+0.5% Molybdenum+0.5% Ceramic Fibers	19.7	350 deg / 3K psi, 1 day, wet					7,148		1,285							

148	5.11	Hybrid Portland	Class H + 35% silica flour+1.0% Kevlar pulp	16.6	350 deg / 3K psi, 1 day, wet					2,899		562					
149	5.18		Class H + 35% silica flour+1.0% 29 Milled fibers	16.6	350 deg / 3K psi, 1 day, wet					6,731		883					
150	5.18		Class H + 35% silica flour+1.0% 38 Milled fibers	16.6	350 deg / 3K psi, 1 day, wet					8,143		1,138					
151	5.19		Class H + 35% silica flour+1.0% Chopped fibers	16.6	350 deg / 3K psi, 1 day, wet					7,642		1,026					
152	4.03	Non-Portland (152 – 188)	Class H + 35% silica flour+10% hexamethyltrisiloxane	16.6	350 deg / 3K psi, 1 day, wet					-		-					
153	2.90		Class H + 35% silica flour+20% hexamethyltrisiloxane	16.6	350 deg / 3K psi, 1 day, wet					-		-					
154	4.38		Class H + 35% silica flour+10% polyethylene oxide	16.6	350 deg / 3K psi, 1 day, wet					3,095		522					
155	4.38		Class H + 35% silica flour+20% polyethylene oxide	16.6	350 deg / 3K psi, 1 day, wet					2,524		484					
156	40.0%		30% MgHPO <sub>4</sub> + 30% AlOOH			Compressives at 350 °F, 3Kpsi, 3day cure					-						
157	40.0%		29.6% MgHPO <sub>4</sub> + 29.6% AlOOH + 3.8% Triethanol-amine			Compressives at 350 °F, 3Kpsi, 3day cure					-						
158	35.0%		32.5% MgHPO <sub>4</sub> + 32.5% Al(OH) <sub>3</sub>			Compressives at 350 °F, 3Kpsi, 3day cure					-						
159	36.5%		43.5% MgHPO <sub>4</sub> + 21.7% Al(OH) <sub>3</sub>			Compressives at 350 °F, 3Kpsi, 3day cure					-						
160	36.5%		47.6% MgHPO <sub>4</sub> + 15.9% Al(OH) <sub>3</sub>			Compressives at 350 °F, 3Kpsi, 3day cure					-						
161	5.0%		47.5% MgHPO <sub>4</sub> + 47.5% Al(PO <sub>3</sub> ) <sub>3</sub>			Compressives at 350 °F, 3Kpsi, 3day cure					65						

162	2.6%	Non-Portland (continued)	48.7% Al(PO <sub>3</sub> ) <sub>3</sub> + 48.7% Al(OH) <sub>3</sub>		Compressives at 350 °F, 3Kpsi, 3day cure					-								
163	3.3%		58% CaO + 38.7% Na <sub>2</sub> SiO <sub>3</sub>		Compressives at 350 °F, 3Kpsi, 3day cure					978								
164	27.8%		16.7% Al(OH) <sub>3</sub> + 33.3% CaO + 22.2% Na <sub>2</sub> SiO <sub>3</sub>		Compressives at 350 °F, 3Kpsi, 3day cure					347								
165	49.0%		20.4% CaOH + 30.6% Na <sub>2</sub> SiO <sub>3</sub>		Compressives at 350 °F, 3Kpsi, 3day cure					300								
166	49.0%		13.5% CaOH + 13.5% Na <sub>2</sub> SiO <sub>3</sub> + 27% AlOOH		Compressives at 350 °F, 3Kpsi, 3day cure					133								
167	45.2%		13.7% CaOH + 13.7% Na <sub>2</sub> SiO <sub>3</sub> + 27.4% MgHPO <sub>4</sub>		Compressives at 350 °F, 3Kpsi, 3day cure					82								
168	48.0%		13% CaOH + 13% Na <sub>2</sub> SiO <sub>3</sub> + 26% Al(PO <sub>3</sub> ) <sub>3</sub>		Compressives at 350 °F, 3Kpsi, 3day cure					166								
169	26.6%		22% CaOH + 33% Na <sub>2</sub> SiO <sub>3</sub> + 18.4% Silica Flour		Compressives at 350 °F, 3Kpsi, 2day cure					398		1,305						
170	34.0%		37.7% Na <sub>2</sub> SiO <sub>3</sub> + 28.3% Alumina							-		-						
171	32.0%		35.5% Na <sub>2</sub> SiO <sub>3</sub> + 26.6% Alumina + 6% MagOx L							-		-						
172	32.0%		35.5% Na <sub>2</sub> SiO <sub>3</sub> + 26.6% Alumina + 6% AlOOH							-		-						
173	29.3%		32.5% Na <sub>2</sub> SiO <sub>3</sub> + 24.4% Alumina + 13.8% Silica Flour							-		-						
174	32.5%		40.5% Na <sub>2</sub> SiO <sub>3</sub> + 27% Alumina							-		-						
175	30.5%		38.1% Na <sub>2</sub> SiO <sub>3</sub> + 25.4% Alumina + 6% AlOOH							-		-						



176	25.2%	Non-Portland (continued)	31.4% Na <sub>2</sub> SiO <sub>3</sub> + 21% Alumina + 4.9% Al <sub>2</sub> O <sub>3</sub> + 17.5% Silica Flour							-		-					
177	32.5%		40.5% Na <sub>2</sub> SiO <sub>3</sub> + 27% CaOH							608		447					
178	30.5%		38.1% Na <sub>2</sub> SiO <sub>3</sub> + 25.4% CaOH + 6% Al <sub>2</sub> O <sub>3</sub>							328		309					
179	28.8%		36% Na <sub>2</sub> SiO <sub>3</sub> + 24% CaOH + 5.6% Al <sub>2</sub> O <sub>3</sub> + 5.6% Alumina							1,133		820					
180	24.0%		30% Na <sub>2</sub> SiO <sub>3</sub> + 20% CaOH + 4.7% Al <sub>2</sub> O <sub>3</sub> + 4.7% Alumina + 16.6% Silica Flour							1,242		971					
181	30.0%		42% Na <sub>2</sub> SiO <sub>3</sub> + 28% CaOH			728		97		780		86					
182	39.6%		37.9% Na <sub>2</sub> SiO <sub>3</sub> + 26.6% CaOH + 5.9% Al <sub>2</sub> O <sub>3</sub>			589		119		850		107					
183	29.2%		34.4% Na <sub>2</sub> SiO <sub>3</sub> + 25.8% CaOH + 5.3% Al <sub>2</sub> O <sub>3</sub> + 5.3% Alumina			651		108		854		205					
184	30.0%		27.6% Na <sub>2</sub> SiO <sub>3</sub> + 18.4% CaOH + 4.3% Al <sub>2</sub> O <sub>3</sub> + 4.3% Alumina + 15.4% Silica Flour			451		113		1,918		216					
185	30.0%		31.5% Na <sub>2</sub> SiO <sub>3</sub> + 21% CaOH + 17.5% SF			51		663		1,072		166					
186	30.0%		29.4% Na <sub>2</sub> SiO <sub>3</sub> + 19.6% CaOH + 4.6% Al <sub>2</sub> O <sub>3</sub> + 16.4% Silica Flour			128		785		1,787		169					
187	30.0%		29.4% Na <sub>2</sub> SiO <sub>3</sub> + 19.6% CaOH + 4.6% Alumina + 16.4% Silica Flour			81		716		799		200					
188	0.0%		62.5% Resin 862 + 37.5% Epicure W (400gms:240gms)														

**Appendix B – DeepTrek Phase II Data**

Data generating tasks associated with Phase II of this project included performing conventional and unconventional tests to confirm large-scale performance, and data associated with the newly-developed test protocols and equipment, including HPHT Annular Seal, HPHT expansion, and Direct Tensile method.

Table B1 shows the results of the candidate systems in the HPHT Annular Seal testing:

<i>Pressure</i>	<i>Baseline 99</i>			<i>Pre-Stressed Cement</i>		
	<i>Test 1</i>	<i>Test 2</i>	<i>Test 3</i>	<i>Test 1</i>	<i>Test 2</i>	<i>Test 3</i>
1,000	4,356	4,356	4,356	15,245	21,779	17,423
2,000	8,712	8,712	8,712	30,491	43,558	34,847
3,000	13,068	13,068	13,068	45,736	65,338	52,270
4,000	17,423	17,423	17,423	60,982	87,117	69,693
5,000	21,779	21,779	21,779	76,227	108,896	87,117
6,000	26,135	26,135	26,135	91,473	130,675	104,540
7,000	30,491	30,491	30,491	91,473	152,454	121,963
8,000	34,847	17,423	17,423	104,540	174,234	139,387
9,000	39,203	19,601	19,601	117,608	196,013	156,810
10,000	21,779	21,779	21,779	130,675	217,792	152,454
<i>Cum</i>	217,792	180,767	180,767	764,450	1,197,855	936,505
<i>Average</i>	193,109			966,270		

**Table B1 – Results of Hard Formation HPHT Annular Seal Testing**

Table B2 shows the result of Hydraulic Bond Testing. This test is distinguished from Mechanical Shear bond by measuring the pressure required to breach the annular seal in a test sample with hydraulic pressure.

<i>Sealant Material</i>	<i>Hydraulic Bond - psi</i>
Baseline 99 Cement	3,800
Pre-Stressed Cement	6,000
Resin	6,425

**Table B2 – Hydraulic Bond Test Results**

Other Phase II data includes the Resin matrix, representing the viable Ultra Seal R formulations tested, as well as formulation and conventional lab testing (thickening time, etc) for both Ultra Seal R as well as PRESTRESSED CEMENT. This data is presented in Tables B3 through B6 below:

Table B3 family - Ultra Seal R Matrix. Values in Comp columns are Component quantity as a percent of base resin.

Table B3a - 80 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
100					1:24	hard
65					1:22	hard
35					2:16	hard
25					3:12	hard
24					4:00	hard
23					5:00	hard
20					5:40	soft
15					6:00	soft
10					7:00	no set
5					8:00	no set
25		10			5:00	soft
30		10			5:00	hard rubber
35		10			5:00	hard rubber
40		10			5:00	hard rubber
45		10			5:00	hard rubber
25		20			7:00	soft
30		20			6:00	soft
35		20			6:20	soft
40		20			5:20	soft
45		20			5:20	hard rubber

Table B3b - 100 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
35					2:00	hard
25					2:20	hard
20					2:40	hard
15					3:00	hard
10					8:00	soft
5					over 8	no set
5	15				over 8	soft
13	5				5:20	hard
15	5				6:00	soft
20	5				6:00	hard
25	5				4:20	hard
25	10				4:00	hard
20		20			7:00	hard
25		20			5:20	hard
25		30			7:00	hard
30		30			5:40	hard
40		30			4:00	hard

Table B3c - 120 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
25					1:40	hard
20					2:00	hard
19					2:40	hard
18					3:00	hard
17					3:20	hard
15					8:00	hard
30	15				2:00	hard
30	10				1:40	hard
30	5				1:40	hard
30	5				2:20	hard
25	10				5:00	hard
25	5				2:00	hard
20	5				5:40	hard
15	10				8:00	hard
10	15				over 8	hard
20		20			5:20	hard rubber
25		20			4:00	hard rubber
20		30			9:00	hard rubber
22		30			8:00	hard rubber
25		30			5:20	hard rubber
27		30			5:00	hard rubber
30		30			4:00	hard rubber
35		30			3:20	hard rubber
40		30			3:00	hard

Table B3d - 140 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
20					1:20	hard
18					1:40	hard
15					2:00	hard
20	5				1:40	hard
20	10				2:00	hard
20	15				2:20	hard
20	20				2:20	hard
20	25				2:20	hard
15	5				2:20	hard
15	15				5:00	hard
15	25				5:20	hard
10	5				4:40	hard
10	15				5:00	hard
10	25				5:20	hard
5	10				12:00	tacky
5	15				12:00	tacky
5	20				12:00	tacky
15		10			3:20	hard
15		20			6:00	hard rubber
25		20			2:20	hard
15		30			9:00	hard rubber
20		30			6:00	hard rubber
22		30			4:00	hard rubber
25		30			3:20	hard
30		30			2:00	hard rubber
35		30			1:40	hard
40		30			1:20	hard

Table B3e - 160 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
15	5				1:40	hard
15	15				2:20	hard
15	25				2:20	hard
15	35				2:40	hard
10	5				3:00	hard
10	15				3:00	hard
10	25				3:20	hard
10	35				3:40	hard
10	40				4:00	hard
10	45				4:00	hard
10	55				4:00	hard
15		10			2:00	hard
15		20			3:40	hard rubber
15		30			6:40	hard rubber
	30				7:40	hard
	35				6:40	hard
	40				6:20	hard
	45				6:20	hard
	50				7:00	hard

Table B3f - 180 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
10	5				2:00	hard
10	15				2:00	hard
10	25				2:00	hard
10	35				2:00	hard
10	40				2:00	hard
15		20			2:40	hard
20		20			1:20	hard
25		20			0:40	hard
30		20			0:20	hard
	15	20			never hard	never hard
	25	20			never hard	never hard
	15				never hard	never hard
	20				7:00	hard
	25				5:20	hard
	30				4:40	hard
	35				4:00	hard
	40				3:40	hard
	45				3:40	hard

Table B3g - 200 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
	20				4:00	hard
	25				3:20	hard
	30				2:40	hard
	35				2:20	hard
	40				2:20	hard
	45				2:20	hard
	20	10			5:40	hard
	25	10			4:20	hard
	30	10			3:40	hard
	35	10			3:40	hard
	20	20			8:40	hard
	25	20			6:00	hard
	30	20			5:00	hard
	35	20			4:20	hard

Table B3h - 220 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
	20	20			6:20	hard rubber
	25	20			4:00	hard rubber
	30	20			3:00	hard
	35	20			2:40	hard

Table B3i - 240 deg F

<i>Comp A</i>	<i>Comp B</i>	<i>Comp C</i>	<i>Comp D</i>	<i>Comp E</i>	<i>Unpump Time</i>	<i>24 Hr Set</i>
	15	20			7:30	hard rubber
	20	20			4:30	hard
	25	20			3:00	hard
	30	20			2:00	hard
	35	20			2:00	hard

Table B4 - Resin Weighout, Thickening Time, and Rheology

100:40:17

Resin: Comp E: Comp B

*Weighout*

Comp	sg	gms	cc	% bwor	150 gm mix
Barite	4.34	0	0	0	0
Resin	1.19	414	348	100	96
Comp E	.96	165.6	173	40	38.4
Comp B	1.02	70.4	69	17	16.3
SF 325 mesh	2.65	0	0	0	0

Thickening Time: at 340 deg F, 1,850 initial, 12,200 psi reached in 65 minutes

14,480 ft Well, 340 deg F BHCT

Test # 100-203

Initial Bc 3

40 Bc 1:25

70 Bc 1:26

Test tubes at 24 hr: Firm / Soft

*Rheology*

Speed	80 deg F	190 deg F
300	190	24
200	130	14
100	68	6
60	40	2
30	20	1
6	4	1
3	2	1

Table B5 – Resin Weighout, Thickening Time, and Rheology

100:40:17:10

Resin: Comp C: Comp B: Comp E

*Weighout*

Comp	sg	gms	cc	% bwor
Comp E	.96	38.9	41	10
Resin	1.19	389	327	100
Comp C	.99	155.6	157	40
Comp B	1.02	66.1	65	17
SF 325 mesh	2.65	0	0	0

Thickening Time: at 340 deg F, 1,850 initial, 12,200 psi reached in 65 minutes

14,480 ft Well, 340 deg F BHCT

Test # 111-389

Initial Bc 5

40 Bc 4:58 hrs:min

70 Bc 5:40 hrs:min

Test tubes at 24 hr: Soft

*Rheology*

Speed	80 deg F	190 deg F
600	248	30
300	130	12
200	86	8
100	44	4
60	24	2
30	12	1
6	2	1
3	1	1
PV	243	
TY	5	



Table B6 – PRESTRESSED CEMENT Weighouts, Thickening Time, and Rheology  
*Weighout*

Comp	Conc	unit	SG	Grams
Lehigh H	94	lb / sk	3.14	546.34
SA-1	.5	% bwoc	.6	2.73
SA-2	25	% bwoc	3	136.59
SA-3	2.5	% bwoc	1.43	13.66
SA-4	30	% bwoc	3.58	163.90
SA-5	.14	gal/sk	1.26	191.22
100 mesh silica sand	35	% bwoc	2.65	8.55
Fresh Water	5	gal/sk	1	242.37

Density: 18.12

Yield: 1.66

*Free Fluid*

Conditioning Time (hr:min):	0:20
Measured (mL):	0
Free Fluid (%):	0

*Stirred Fluid Loss*

Conditioning Time (hr:min):	0:20
Test Temp (deg F):	230
Collected Fluid (mL):	17
Time (min):	30
API FL (mL/30 m):	34

*Thickening Time*

Test #	100-393	100-582
Test Temp (deg F)	230	230
Time to Temp (min)	68	68
Initial Pressure (psi)	825	825
Final Pressure (psi)	9825	9825
Initial Bc	27	16
40 Bc (hr:min)	3:06	3:24
70 Bc (hr:min)	3:37	3:48

*Rheology*

Speed	80 deg F	190 deg F
300	366	132
200	254	88
100	130	42
60	78	22
30	38	10
6	6	2
3	4	1
PV	354	135
TY	12	-3

## Appendix C – Ultra Seal-R Field Test Details and Results

### Stone Energy 210 Degree Pumping

Although there is no formal job report for this job, it represents the deepest coil tubing job performed to date. BHCT was 180 deg F, and depth was 9,000 ft. This job illustrates the ability to control Ultra Seal-R in extreme conditions. The material set as designed, and the job was run successfully.

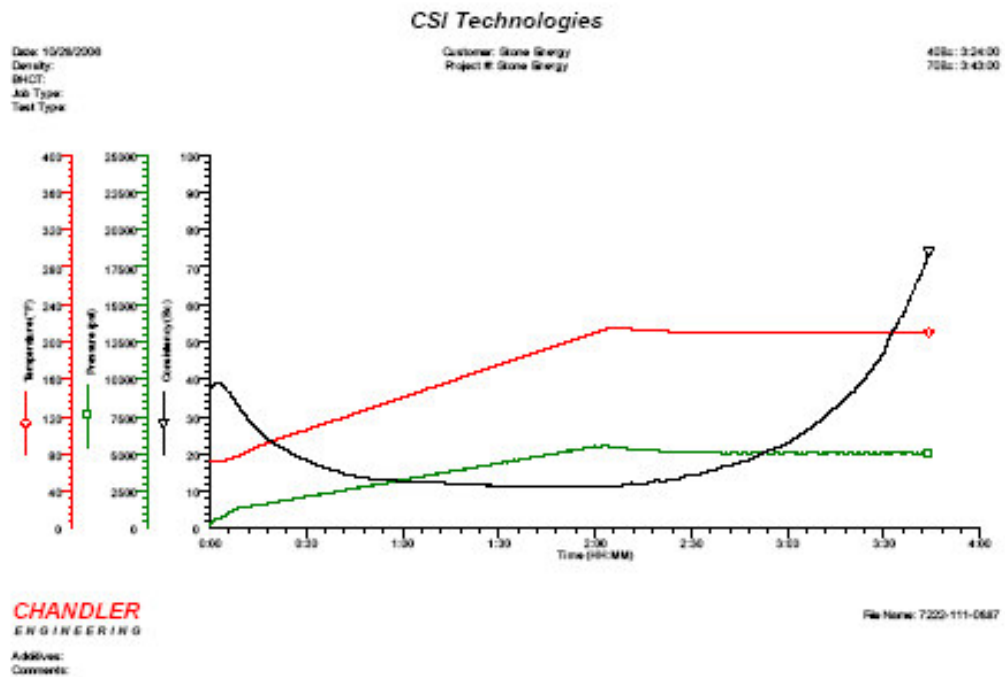
Perform thickening time and place cubes in oven at 210°F and 160°F

Test#111-887

36 Bc Initial

40 Bc 3:24 hrs:min

70 Bc 3:43 hrs:min



24hr cubes at 160°F were rubbery hard, cubes at 210°F were rock hard  
48hr cubes at 160°F were still rubbery hard.

### 295 degrees F Dump Bailer Formulation

The following two formulations were developed for customers for potential jobs at 295 deg F and 340 deg F. For various reasons unrelated to the fluid design the jobs were never performed. The lab test results show that the material is viable up to 340 deg F, and continuing development work is still underway to increase the temperature range as well as improving the properties of the materials engineered for higher temperatures.

#### 295 Degree Dump Bailer

Material	Density	sg	Design	% by Weight	Density (lb/gal)
Resin	9.9ppg		1.19	100	0.524
Component A	7.5ppg		0.9	20	0.105
Component B	7.8ppg		0.94	0	0.000
Component C	8.5ppg		1.02	13	0.068
Component D			4.33	58	0.304
Total Resin Volume (gal)					(mL)
0.100					378.540
Total Resin Weight (lb)					(g)
1.202					545.089

#### Recipe

Material	Weight (lb)	Weight (g)
Resin	0.63	285.39
Component A	0.13	57.08
Component B	0.00	0.00
Component C	0.08	37.10
Component D	0.36	165.52
Component E as needed		

340 degrees F Dump Bailer Formulation

E00059-22

Rheologies

100:40:17:10  
Resin:Comp C:Comp B:  
Comp E

	80°F	190°F
600rpm	248	30
300rpm	130	12
200rpm	88	8
100rpm	44	4
60rpm	24	2
30rpm	12	1
6rpm	2	1
3rpm	1	1
PV	243	
TY	5	

Comp E
Resin
Comp C
Comp B
SF 325 mesh

sg	gms	cc	%bwt
0.98	38.8	41	10
1.19	388	327	100
0.99	165.8	157	40
1.02	88.1	85	17
2.65	0	0	0

total	648.8	569	sg	Density
			1.10	9.18

Thickening Time: at 340°F with 1,850psi initial,  
12,200psi reached in 65minutes

14480ft Well, 340°F BHCT

Test # 111-389  
Initial B<sub>i</sub> 5  
40 B<sub>c</sub> 458 70 B<sub>c</sub> 540

	24hr
Place sample in test tubes at 340°F	soft

### *Ultra Seal-R Job Report*

The following job reports represent a selected Ultra Seal-R job report conducted during Phase III of this project. Many more jobs were performed, but this job illustrates the capability of the material as well as the field procedures employed in application.

The most unusual aspect of this job was the requirement to pump Ultra Seal-R over 9,000 ft down 1/4" coiled tubing. High friction pressure and the resultant heat generation in the material created unique engineering requirements. Additionally, the low pump rate required special pumps and significant thickening time. In spite of the difficulties encountered, the job was completed successfully.

UltraSeal-R Job Report Log



Job Report **UltraSeal**

Company / Customer BP		CSI Consultant R. Stanford? D. Brown		CSI Project No. P00056		Job Type UltraSeal	
Well Location / Block / Number Grand Isle 40 well 1-07				Cementing Service Company Superior Energy Services		Rlg Name L/B Vision	
<b>WELLBORE GEOMETRY</b>							
<b>Work String (upper)</b>			<b>Work String (lower)</b>			<b>Open Hole</b>	
OD, in	Weight, lb/ft	ID, in	OD, in	Weight, lb/ft	ID, in	OD, in	MD, ft
1/4			2 3/8	4.85			
<b>Casing / Liner</b>			<b>Perforations - Upper Set</b>				
OD, in	Weight, lb/ft	Grade	ID, in	MD, ft	TVD, ft	Top of Liner, ft	Top Perf, ft
7 5/8	26.40	N-80		9,242			Bottom Perf, ft
<b>Previous Casing / Liner</b>			<b>Perforations - Lower Set</b>				
OD, in	Weight, lb/ft	Grade	ID, in	MD, ft	TVD, ft	Top of Liner, ft	Top Perf, ft
							Bottom Perf, ft
<b>FLUIDS INFORMATION</b>				<b>JOB PARAMETERS / CALCULATIONS</b>			
Wellbore Fluid		Description	Den, lb/gal	Vol, bbl	Calculated % OH Excess:		
Spacer / Flush				-	Displacement Pumped By:		
Displacement					Desired Squeeze Pressure, psi		
					Final Squeeze Pressure, psi		
<b>Lead Cement</b>			<b>Tail Cement</b>				
Cement Type			Cement Type			Lead Volume, sack	
Density, lb/gal			Density, lb/gal			Lead Volume, bbl	
Yield, ft <sup>3</sup> /sk			Yield, ft <sup>3</sup> /sk			Lead Mix Water, bbl	
Mix Water, gal/sk			Mix Water, gal/sk			Lead CSI TTT, hr:min	
Mix Water Type			Mix Water Type			Lead Batch Mixed?	
Total Fluid, gal/sk			Total Fluid, gal/sk			Lead SRT, minute	
Additives	Conc	Units	Additives	Conc	Units	Tail Volume, sack	
						Tail Volume, bbl	
						Tail Mix Water, bbl	
						Tail CSI TTT, hr:min	
						Tail Batch Mixed?	
						Tail SRT, minute	
<b>DOWNHOLE TOOLS</b>							
Squeeze Tool Type:				Other DH Tool(s):			
Squeeze Tool Brand:				Was a Squeeze Manifold Used?			
<b>ON-SITE SERVICE TIME / MILEAGE</b>							
Product Code	Description	Qty	Qty	Unit Price	Amount		
CSITU2005	Travel To / From Location, per man, per day	2	2	\$ 1,250.00	\$ 5,000.00		
CSITU2000	Consultant On-Site, per man, per day	2	9	\$ 1,650.00	\$ 29,700.00		
CSITU2001	Consultant Standby, per man, per day			\$ 1,250.00	\$ -		
CSITU2030	Mileage to/from location, per mile		800	\$ 2.25	\$ 1,800.00		
<b>Total:</b>					<b>\$ 36,500.00</b>		
Called Out:	08 Jul 16:00	CSI Project No.		Company Rep. Signature:			
On Location:	09 Jul 3:00	P00056					
Released:	18 Jul 1:00						







Grand Isle 40 well 1-07		JOB LOG				CSI Project No. - P00056
Date dd Month	Time hr:min	Density lb/gal	Rate bpm	Pressure psi	Volume bbl	Event
10	Jul					Waiting on resin to set up( which has already setup)
						Coil tubing working on another well-setting cement plug
						Coil tubing freewheeled causing birdnest on spool
						Waiting to get another empty reel to POOH
11	Jul					Standing-by to change out reels.
12	Jul					Standing-by
13	Jul					Standing-by
14	Jul					Standing-by
15	Jul					Standing-by
16	Jul					
17	Jul					Cut tubing
	11:00					
	14:00					Rig up CT
	15:00					St. in hole w/ CT
	19:23		0.5	4,700		Bk. Cir. Down CT and tbg.
	19:28					Close tubing annulus-Bk. Circ. Down CT and 75/8 casing 1/4 bpm=3250 1/2bpm = 5750
	19:50					Mix Ultra-Seal R
						Part A + 880# barite- roll for 10 mins.
	20:25					Part B
	20:40		0.3	7,700	3.0	Start pumping resin down coil tubing
						excessive pressure in coil- pump as slow as possible
	20:52					Pump 2 bbls sw behind resin to clear lines
	20:55					Switch to sw tank -pump remaining 14.5 bbls.
	21:20					After pumping 10bbls. Of the 14.5 pressure dropping once resin cleared spool.
	21:30					CT cleared- shut down- pull 500ft. w/o circ.
						Start circ. CT @ 1/4 bpm while pulling out of holw
	23:45					Out of hole- clean blender, pumps,& CT w/ methanol
18	Jul					Released
	1:00					
	2:30					At dock

### *Appendix D – PRESTRESSED CEMENT Field Test Details and Results*

No jobs have been performed with PRESTRESSED CEMENT as of the date of this report. The material is not appropriate for all primary jobs, requiring competent formations or pipe-in-pipe jobs for best results. Several jobs were scheduled during Phase III, but were cancelled for various reasons. The complete job engineering was completed for these jobs, including cement design, thickening time, and rheology. These lab design details are presented in this section. CSI is continuing to market this product with select customers.

Both jobs were designed for Goldking Operating, and for two different wells. The first job was for the Talk #2 well, and the design was executed in March, 2007. The job was a squeeze job at 10,836', with a 214 deg F BHCT and 251 deg F BHST. The second job was designed as a tail-in slurry for the Cobb #1 well. Depth was 17,800', and temperatures (deg F) were 271 circulating and 328 static. Both slurries were designed meeting all cement design parameters for thickening time, rheology, free water, and settling.

A full scale mixing of the PRESTRESSED CEMENT was conducted at a yard facility. A report detailing the job is included following the lab design reports.

PRESTRESSED Cement Design 1



**B01276-2E  
Tail System**

**Laboratory Cement Test Report**

Project No:	B01276 - 2E	Depth MD (ft):	10,836	Test Date:	March 22, 2007
Company:	Goldking Operating	Depth TVD (ft):	10,836	Job Size / Type:	2 7/8 (in) Squeeze
Requestor:	Allen Faircloth	BHST (°F):	251	Well Fluid Density (lb/gal):	8.4
Well Name:	Talk #2	BHCT (°F):	214	Well Fluid Type:	Field Brine
Rig Name:	Key Energy 1401	Temp. Grad. (°F/100ft):	1.58	Test Schedule:	Squeeze - 9.31
		Test Pressure (PSI):	8,130		

**Cement Slurry Design**

Cement Blend	Sack Weight, lb	% of Total Sack Weight	Prod Weight, lb/sk	CSI Log #	
Cement - Class H	94	100	94.00	C 625-A	
Mix Water	Concentration	Units	CSI Log #		
Deionized Water	5.023	gal/sk	Lab Stock		
Function	Additive	Concentration	Units	CSI Log #	Slurry Density (lb/gal):
Silica	Silica Sand	35.000	%bwoc (DB)	C 625-F	<u>18.1</u>
Expansion Agent	MAG-OX	30.000	%bwoc (DB)	Lab Stock	Slurry Yield (ft <sup>3</sup> /sk):
Micro Fine Cmt	Micro Matrix Cement	25.000	%bwoc (DB)	Lab Stock	<u>1.66</u>
Dispersant	NC-S-1	2.500	%bwoc (DB)	C 625-H	Total Mixing Fluid (gal/sk):
Fluid Loss	FL-17	0.300	%bwoc (DB)	C 625-D	<u>5.02</u>
Retarder	PCR-3	1.000	%bwoc (DB)	C-626-D	
Retarder	PCR-4	1.000	%bwoc (DB)	C-626-E	

**Test Results**

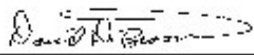
<b>Desired Thickening Time</b>	<u>2 1/2 - 3 Hours</u>				
<b>Total Thickening Time - Cement</b>	40 Bc	70 Bc			
BHCT (°F):	214	2:10	2:16	hrs:mins	
<b>Desired Fluid Loss</b>	<u>150 - 350 mls</u>				
<b>Stirred Fluid Loss</b>					
Test Temp (°F):	214		Conditioning Temp. (°F):	190	
Collected Fluid (ml):	22		Test Angle:	Vertical	
Collection Time (min):	22		Measured Free Fluid (ml):	0.0	
API Fluid Loss (ml/30min):	-		Free Fluid (%):	0.0	
Calculated API (ml/30min):	<u>51</u>				

**Compressive Strength**

Test Temperature (°F):	251	
50 psi at	20:44	hrs:mins
500 psi at	21:47	hrs:mins
- psi at	12:00	hrs:mins
1,887 psi at	24:00	hrs:mins

**Rheological Properties**

Fluid / Mixture	Temp °F	300	200	100	60	30	6	3	PV	YP
Cement	80	440	308	166	106	53	16	8	427	20
100 %	190	248	170	88	52	26	6	4	247	4
									cP	lb/100ft <sup>2</sup>

Project Coordinator:   
David Brown

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B01276, 10836, Squeeze, Cement - Class H, 18.1 lb/gal, Silica Sand, MAG-OX, 214.353057313419F

PRESTRESSED Cement Design 2



PreStressed  
Cement

**Laboratory Cement Test Report**

Test Date: <u>May 31, 2007</u>	Depth MD (ft): <u>17,800</u>	Job Size / Type: <u>2 7/8 (in) Casing</u>
Project No: <u>D00014 - 6</u>	Depth TVD (ft): <u>17,700</u>	Spacer Density (lb/gal): <u>14.0</u>
Company: _____	BHST (°F): <u>328</u>	Spacer Type: <u>Ultra Spacer</u>
Requestor: _____	BHCT (°F): <u>271</u>	Well Fluid Density (lb/gal): <u>13.0</u>
Well Name: _____	Temp. Grad. (°F/100ft): <u>1.4</u>	Well Fluid Type: <u>Water Base Mud</u>
Rig Name: _____	Test Pressure (PSI): <u>12,960</u>	Test Schedule: <u>Casing - 9.10</u>

**Cement Slurry Design**

Cement Blend	Sack Weight, lb	% of Total Sack Weight	Prod Weight, lb/sk	CSI Log #	
Cement - Class H	94	100	94.00	Lafarge	Slurry Density (lb/gal): <u>16.5</u>
Mix Water	Concentration	Units	CSI Log #		Slurry Yield (ft <sup>3</sup> /sk): <u>1.99</u>
Deionized Water	7.323	gal/sk	lab		
Function	Additive	Concentration	Units	CSI Log #	Total Mixing Fluid (gal/sk): <u>7.70</u>
Micro Fine Cmt	MC-500	25.000	%bwoc (DB)	R-566 A1	
Sand	Silica Sand	35.000	%bwoc (DB)	lab	
Dispersant	CD-690	0.750	%bwoc (DB)	R-552 G	
Bonding Agent	MagOx H	30.000	%bwoc (DB)	lab	
HT Susp Aid	HTSA-400	0.400	%bwoc (DB)	R-552 F	
Fluid Loss	CFL-40	1.500	%bwoc (DB)	R-552 D	
Retarder	CR-350L	0.240	gal/sk	R-552 K	
Retarder	CR-430IL	0.120	gal/sk	R-552 L	
Defoamer	CAF-100	0.020	gal/sk	R-502 B	

**Test Results**

**Total Thickening Time - Cement**

BHCT (°F): <u>271</u>	40 Bc	<b>70 Bc</b>	hrs:mins
	4:36	<b>4:57</b>	

**Stirred Fluid Loss**

Test Temp (°F): <u>271</u>
Collected Fluid (ml): <u>22</u>
Collection Time (min): <u>30</u>
API Fluid Loss (ml/30min): <b>44</b>

**Free Fluid**

Conditioning Temp. (°F): <u>190</u>
Test Angle: <u>Vertical</u>
Measured Free Fluid (ml): <u>0.0</u>
Free Fluid (%): <u>0.0</u>

**Rheological Properties**

Fluid / Mixture	Temp °F	300	200	100	60	30	6	3	PV	YP
Cement	80	216	150	78	50	26	6	2	211	8
100 %	190	80	54	30	18	10	2	1	77	3
									cP	lb/100ft <sup>2</sup>

Project Coordinator: Paul Somnier  
Paul Somnier

The above data is supplied solely for informational purposes. CSI Technologies makes no guarantees or warranties, either expressed or implied, with respect to the accuracy or interpretation of this report. The results of this report are provided "as is" based on the information provided.

*Field Mixing Study for PRESTRESSED Cement Report*



**DEPARTMENT OF ENERGY**

**Field Mixing Study for  
Pre-Stressed Cement System**

Prepared For: Roy Long  
Date: July 25, 2007

Prepared by: David Brown  
Sylvester Auzenne

CSI Technologies makes no representations or warranties, either expressed or implied, and specifically provides the results of this report "as is" based upon the provided information.

## **Objective**

The objective for this project was to determine the mixability of the 17.0 lb/gal Pre-Stressed cement slurry with standard field mixing equipment. This investigation involved the use of a re-circulating mixer, commonly used to mix cement slurries in the field to see if any problems or inconsistencies are encountered.

## **Conclusions**

The Pre-Stressed cement blend was mixed at the desired density without encountering any negative mixing issues. The system was easily mixed and demonstrated the ease of pumping in any field situation. All post-job lab testing indicated a successful blend and matched all pre-job lab testing.

## **Laboratory Test Methods**

Laboratory pre-job testing was conducted to establish a control and ensure that a good cement composition was used in the field mixing trial. It involved slurry viscosity measurements, mixing method, free fluid, and fluid loss testing. Laboratory post-job testing will be to perform rheologies, free fluid and fluid loss tests on the samples taken from the field trial.

## **Field Trial Procedure**

Advanced Oilwell Services agreed to perform the field mixing trial at their yard in Brookshire, Texas as seen in Figure D1. They provided the cement from their bulk facility and agreed to dispose of the slurry after mixing and completion of all yard testing. A total volume of 50 sacks (16.65 bbls) of cement was mixed in the re-circulating mixer to simulate a field cementing application. AOS provided a Service Supervisor and an Operator for equipment operation and mixing, while CSI Technologies provided a manager and technical staff to oversee the mixing process and conduct sample collecting and testing.

- Upon arrival to the bulk plant the add mix tank was inspected for any previous job additives to ensure no contamination of cement blend.
- The calculated amount of class H Lehigh cement was added to the scale tank and the weight verified. All of the dry additives were loaded into the add mix tank and then to the scale. The weight was verified before boxing cement four times and then blown to bulk truck.
- The first 6 barrels of slurry consisted of continuous mixing to a density of 17.0 lb/gal as seen in Figure D2. At this point, a sample was taken, the density measured as seen in Figure D3, and Rheologies as seen in Figure 4 were performed. The slurry was mixed continuous in the re-circulating mixer until all 16.5 barrels were mixed. Rheology testing and density checks were taken at various intervals.

The slurry displayed no signs of potential mixing difficulties that would hinder the ability of the cement to be mixed in a field environment.

### Discussion of Results

**Cement Design:** 50 sks Texas Lehigh Class H + 0.5% bwoc Fluid Loss Additive, 25% Micro cement slagbased, 2.5% Dispersant, 30% Magnesium Oxide H, 35% 100 Mesh Silica Sand, mixed at a density of 17.0 lb/gal.

For Table D1 all rheological testing was performed at ambient temperature. Samples were taken from the mixing tank and measured for density and rheology onsite. The final density of 17.0 lb/gal and rheologies listed are comparable to the pre-job testing as seen in Table D2.

Table D1: Rheologies at test site

Sample Number	Time	Density lb/gal	300 RPM	200 RPM	100 RPM	60 RPM	30 RPM	6 RPM	3 RPM
1	Initial	16.6	52	36	18	12	6	2	2
2	5 minutes	16.8	52	34	18	12	6	2	2
3	11 minutes	17.0	70	46	24	16	10	2	1

Table D2 lists the pre-job lab testing along with the post-job lab testing with two separate field blend samples. Measured density, rheologies, free fluid and fluid loss were all comparable.

Table D2: Post Job lab testing at 80°F

Tests		Pre-Job Test (Lab Materials)		Field Blend Sample #1		Field Blend Sample #2	
Measured Density		17.0 lb/gal		17.0 lb/gal		17.0 lb/gal	
Rheology		Initial	80°F	Initial	80°F	Initial	80°F
	300 rpm	80	100	100	110	76	104
	200 rpm	54	68	68	74	52	70
	100 rpm	28	40	36	38	26	38
	60 rpm	18	26	24	26	18	24
	30 rpm	8	14	12	12	8	14
	6 rpm	2	4	4	4	4	6
	3 rpm	1	2	2	2	2	4
	PV	72	90	96	108	75	99
	YP	2	10	4	2	1	5
Fluid Loss		24 ml/30min		28 ml/30min		16 ml/30min	
Free Fluid		0.0%		0%		0.0%	



Table D3 lists a pre-job design along with a field blend sample test. The systems were designed for a simulated well at 18,000 ft with 300°F circulating temperature. The measured density and rheologies were very comparable as well as the fluid loss, free fluid and thickening time.

Table D3: Post Job Lab testing at elevated temperatures

Tests		Pre-Job Test (Lab Materials)		Field Blend Sample #1	
Measured Density		16.5 lb/gal		16.5 lb/gal	
Rheology		Initial	190°F	Initial	190°F
	300 rpm	170	66	154	68
	200 rpm	102	40	126	42
	100 rpm	44	30	56	24
	60 rpm	24	12	34	16
	30 rpm	12	6	18	8
	6 rpm	4	4	4	6
	3 rpm	2	2	2	6
	PV	189	54	147	66
	YP	-19	12	7	2
Fluid Loss at 300°F		75ml/30min		62 ml/30min	
Free Fluid		0.0%		0.0%	
Thickening Time at 300°F (hrs:min)		40 Bc = 6:07 70 Bc = 6:15		40 Bc = 5:07 70 Bc = 5:11	

Figure D1: AOS yard facility



Figure D2: Pre-Stressed Cement slurry in mixing tank



Figure D3: Pressurized Mud Scale for measuring Density



Figure D4: Rheometer for measuring rheologies



## *Appendix E – Abstract and Job Reports Submitted for Hart E&P’s Meritorious Award for Engineering Innovation*

### *Ultra-Seal R Abstract*

#### Introduction:

Ultra-Seal R wellbore sealant is an epoxy product, formulated and applied to seal wellbores as an alternative to Portland cement. While the natural density of the material is slightly greater than water, it can be weighted as desired with no affect on performance. Ultra-Seal R does not readily mix with water, nor is it affected by water contamination, allowing for innovative placement possibilities in difficult conditions. The material set can be controlled from 40 deg F. to 350 deg F. Benefits of the material as a wellbore sealant include: improved strength, improved bond strength, non shrinking, and ability to invade permeable solids and harden.

#### Background:

The material was originally formulated for a non-oilfield application involving electrical insulation at high temperatures. The material possessed intriguing mechanical properties as well as tolerance to aqueous fluids, leading to a laboratory testing program to determine potential as a wellbore sealant. The material was adapted to accommodate mixing and placement constraints associated with wellbore sealants. The basic components are a resin and a hardener, although other materials are added to tailor properties and pump time as desired. These materials include diluents, lightweight additives, heavy weight additives, and there are also alternative hardeners available for different temperature ranges.

#### Mixing and Pumping:

Ultra-Seal R resin and hardener are mixed on the surface in conventional mixing equipment. Cleanup is effectively accomplished with a minimal quantity of a methanol and water mixture. To date, the material has been primarily batch-mixed, although a continuous mixing method is achievable. The components of Ultra-Seal R are two liquid components, allowing for a more precise mixing methodology than conventional Portland cement. There are moderate personal protective equipment requirements when handling Ultra-Seal R, and are directed toward prolonged respiration of vapors and skin contact.

#### Placement:

The ability of Ultra-Seal R to tolerate water without mixing with it allows for innovative placement methods and wellbore solutions. The material can be weighted to fall though standing water in the wellbore, or lightened to float on top. Because there are no solids in the basic material, it can be effectively squeezed into casing leaks or into formations as a solids-free liquid and then harden in place. Ultra-Seal R can be used to create virtual wellbores, in which the resin penetrates into the formation, to consolidate weak or damaged zones. Other innovative solutions include gravel pack remediation. Placement may be accomplished by circulating down coiled or jointed tubing, bull heading, gravity feed, dump bailer or controlled volume coiled tubing injection.

#### Properties:

Ultra-Seal R can be tailored to a wide variety of wellbore conditions, both in terms of temperature, depth, and wellbore fluid interaction. When set, the material is impervious to essentially all wellbore fluids and gasses. The set monolith typically has the consistency of a hard, tough plastic. However, the material can be formulated to have varying degrees of hardness, ranging from that of a hard plastic to a stiff rubber. Testing has shown that Ultra-Seal R, unlike Portland cement, is extremely ductile in its set phase, meaning that the material deforms without fracturing when loads are applied. Further, when loads are released, the material rebounds to achieve its initial shape. In the wellbore environment, this characteristic offers the ability to absorb a great deal of stress without fracturing and creating a flowpath for wellbore fluids (seal failure). Ultra-Seal R is readily drillable and millable utilizing conventional bits. Exact strength measurements are difficult to obtain due to the unique ductility of the material. Application of conventional cement lab test methodologies have yielded high compressive and tensile strengths, although it is difficult to pinpoint the point of failure due to the non-brittle behavior of the material. . Compressive strengths of 5,500 psi, tensile strengths of 3,700 psi (Splitting Tensile Test), and mechanical shear bonds of 1,900 psi have been observed in the lab.

#### Field History:

Ultra-Seal R has been applied in over 100 jobs, ranging from P&A to Squeeze work, at a variety of temperatures up to 250 deg F. Normally, the material is used to fix problems after application of conventional Portland cements have failed. Ultra-Seal R has been used successfully in rigless situations due to its ability to fall through wellbore fluids, saving the customer rig costs and time. When sealing gas migration between casing strings, the high sealing efficiency allows for smaller material volume than jobs performed with Portland cement. This means smaller milled windows and reduced rig time while providing a successful seal. Pre-engineered kits have also been developed and applied for dump bailer work. Because of smaller volumes required to achieve a seal and the ability to achieve a quick set, the amount of time required to perform these jobs is dramatically reduced, and the pressure test can be much higher than with Portland cement.

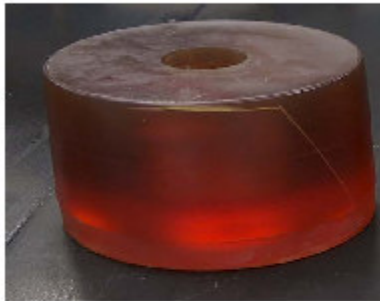
#### **Job Report:**

A customer was abandoning a well, and having difficulty, due to the inability of conventional Portland cement squeezes to seal the perforations in the production zone. Perforations were to be squeezed at 10,196 ft and 250 degrees F. 5 bbls of Ultra-Seal R were weighted to 16.5 lbs/gal, and bullheaded down the production tubing while holding pressure on the backside. The job was mixed and placed, with 3 bbls entering the perfs and formation, leaving two bbls in the production tubing. After the Ultra-Seal R was pumped and displaced, 2,100 psi were held on the tubing and 2,050 psi on the tubing-casing annulus for 14 hours. Following the WOC time, the tubing was successfully pressure tested to 2,000 psi, and the job declared successful.



# ULTRA SEAL®-R (LIQUID BRIDGE PLUG™)

M&D Industries of Louisiana, Inc.



MAY 2005

ULTRA SEAL®

Case Histories

SOUTH PASS 49 2

OCS-G 3206 B-1

## CASE HISTORY-CHEVRON USA

### OBJECTIVE:

To seal micro annular gas migration between the 20" X 30" casing strings and pump the surface cement plug as per MMS requirements for P&A.

**USING ULTRA SEAL®-R SAVED THE OPERATOR FROM HAVING TO REMOVE ANY OTHER CASING STRINGS TO SET AN INFLATABLE PACKER.**

### *Special points of interest:*

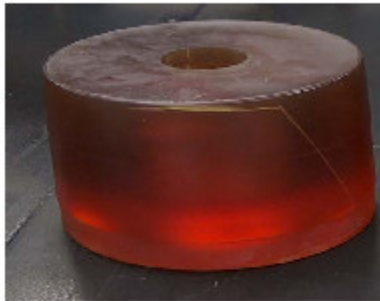
Prior to the ULTRA SEAL®-R treatment, a 30' window was planned to be milled through the 10-3/4" and 20" casing out to the 30". Due to mechanical problems only 20' of the 20" was able to be milled out to the 30". It was decided to place sand followed by ULTRA SEAL®-R up into the 10-3/4" casing.

The BHT at plug depth was estimated to be 45-50 degrees F.

- NON-SHRINKING
- FUNCTIONS AT LOW TEMPS
- OVER 10 TIMES THE SHEAR BOND OF CEMENT
- IMPERMEABLE TO GAS
- ULTIMATE COMPRESSIVE STRENGTH OF OVER 6,000 PSI

# ULTRA SEAL®-R (LIQUID BRIDGE PLUG™)

M&D INDUSTRIES OF LOUISIANA, INC.



JUNE 2005

ULTRA SEAL

800-772-6833

Vermillion 31

OCS-G 2868 #3

## CASE HISTORY-CHEVRON USA

### OBJECTIVE:

The operator wanted to seal a leaking packer in an offshore production well without the use of a rig due to availability and pricing. Engineering wanted a fluid that would fall through sea water in the vertical 7 X 3 1/2 inch annulus. The fluid required an extended set time while maintaining its properties and forming an effective seal.

BHT: 205° F

Completion Fluid:  
8.6# Inhibited SW

### APPLICATION:

A special formulation of ULTRA SEAL®-R was designed with additional density to increase the settling rate.

After mixing 168 gallons of ULTRA SEAL®-R, the resin sealant was loaded into the annulus and chased with 9 bbls of sea water. The well was shut in and the resin sealant was allowed to fall through the sea water for 14 hours to settle on top of the packer.

### RESULTS:

After a twenty four hour cure time the ULTRA SEAL®-R plug was tested to 1,000 psi for 30 minutes with no recorded pressure losses.

The Operator was pleased with the total operation and that a rig-less operation had been performed that saved thousands of dollars in rig time not to mention any lost production waiting on a rig.

### *Special points of interest:*

- NON-SHRINKING
- FUNCTIONS AT LOW TEMPS
- OVER 10 TIMES THE SHEAR BOND OF CEMENT
- IMPERMEABLE TO GAS
- ULTIMATE COMPRESSIVE STRENGTH OF OVER 6,000 PSI

## ULTRA SEAL R DUMP BAILER KIT

Ultra Seal R is a special high bond sealant tailored for oil well applications. A new dump bailer kit has been developed that produces maximum attainable down-hole shear bond/sealing capabilities when compared to anything currently available on the market. Different accelerators and hardeners have been developed to allow for product placement and strength development over a wide temperature range. The dump bailer kits are designed to produce a solid plug, with extremely high shear, just 12 hours after application under any bottom-hole temperature up to 350°F.

This equates to increased job efficiency that will result in less fill requirements for a given application and decreased job time. The Ultra Seal R dump bailer kit is an excellent choice when height/shear bond requirements are tough and job time/cost is important.

**Key Features Include the Following:**

- Maximum Shear Bond to Casing/Tubing
- Excellent Gas Migration Control
- Ease of Mixability
- Wide Temperature Application
- 10+ X Shear Bond of Cement
- Non-Shrinking
- Saves Time and Cost



The new Ultra Seal R dump bailer kit provides a pre-engineered kit that eliminates the need for laboratory testing and design for each application. Each kit comes with easy to follow instructions for mixing and application.

Generic Condition	Typical Dump Bailer (2-inch)	Ultra Seal R Dump Bailer (2-inch)	Typical Dump Bailer (1.5-inch)	Ultra Seal R Dump Bailer (1.5-inch)
Bottom-Hole Temperature	200°F	200°F	275°F	275°F
Casing Size	5-inch	5-inch	2 7/8-inch	2 7/8-inch
Required Plug Length	20-ft	5.5-ft	30-ft	5-ft
Maximum Pressure Test	4,000 PSI	11,000 PSI	10,000 PSI	17,000 PSI
Time for Operation	32+ Hours	12 Hours	29+ Hours	12 Hours

The table above illustrates the amount of time and cost that can be saved when the new Ultra Seal R dump bailer kit is used instead of a typical cement kit. In both instances, the Ultra Seal R dump bailer kit requires less product volume (required plug length), which means fewer dump bailer runs and lower costs, and still results in a larger differential pressure capabilities.



*Appendix F –Technical Transfer*

*2007 IADC / DEA Workshop Presentation*

This project was presented at the 2007 IADC / DEA Workshop. The presentation in its entirety is included in this section. Following the presentation slides, a summary of the Q&A session is detailed.





**CSI Technologies**

## Project Work Team

- CSI Technologies LLC
- Material Manufacturers
- → *Steering Committee*
- → *Operators*



**CSI Technologies**

## Project Objectives

- Determine the cement system properties that affect cement sealing integrity
- Use recently-developed laboratory methods to determine key properties in candidate materials
- Develop Supercement systems(2)
- Test application in wells
- → *Field test including “deep hot” wells* ←
- Commercialization



**CSI Technologies**

## Problem: Long Term Zone Isolation in HPHT Wells

- High Temperature and Pressure
- Deviation angles - Placement is difficult
- High Density systems – 17 to 20 ppg well fluids
- High Pressure Gas / Gas migration
- Narrow Annuli / High Friction
- Liners versus longstrings
- Tie Backs and Expandable Liners
- CO<sub>2</sub> and H<sub>2</sub>S common



**CSI Technologies**

## Phase I Work Plan

- Literature search - Portland cements & Non-Portland cements
- Low temperature material property screening
- High temperature material property screening
- Unconventional material tests



## Phase I Work Results

- Identified 5 system types for further investigation
- 2 Non-Portland systems
- 3 Portland-based systems
- Reactive fiber reinforcement as augment to both Portland and non-Portland systems



## Phase II Work Plan

- Narrow potential systems to two
  - Resin
  - Pre-stressed cement
- Manufacture Supercement to specification
- Conventional and Nonconventional Batch testing to confirm performance on commercial scale
- Evaluate performance in large scale mixing, shearing, and drillout tests



## Phase II Work Plan

- Design two new test apparatus
  - Direct Tensile Test
  - HTHP Annular Seal Test
- Rationale
  - No standard tensile test; splitting tensile results difficult to interpret
  - Unconstrained tensile strength for Expanding cements
  - HTHP sealing performance critical



## Phase II Results

- Resin:
  - Significant formulation production, testing, and property tailoring
  - Resin kinetics for low and high temperature applications
  - Design program developed
  - Large-scale mixing tests conducted
  - Drillout tests conducted successfully
  - Multiple successful field trials
- Pre-stressed Cement
  - Currently available in commercial field quantities
  - Field-ready slurry has been designed

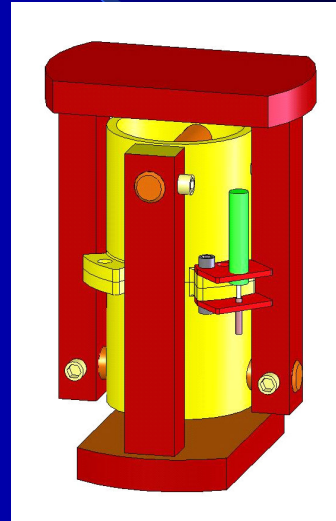


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## Phase II Extension

### Direct Tensile Test

- Utilize compression test machine
- Measure tensile deformation for calculation of Tensile YM
- Perform tensile fatigue

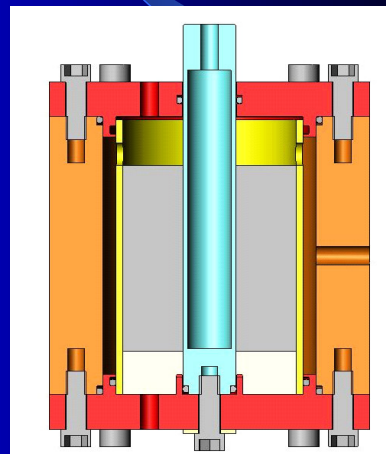


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## Phase II Extension

### HHP Annular Seal

- Measure resistance to annular gas flow at HHP conditions
- Designed for 3,000 psi and 300 deg F
- Measures true constrained tensile strength
- Sealing performance results correlated to energy applied





## Phase II Results

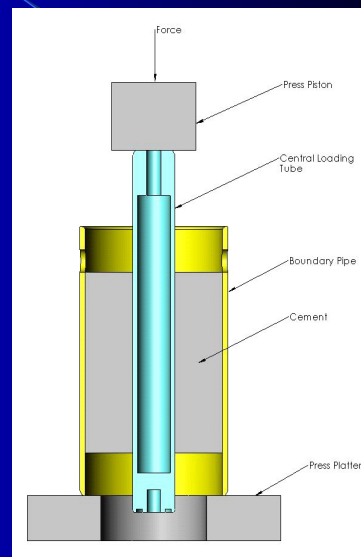
- All test apparatus in service
- HTHP Annular Seal results
  - Ultimate laboratory performance test
  - Baseline: state-of-the-art HTHP Cement: Portland + 35% Silica
  - Resin: Baseline energy to failure \* 100
  - Super Expansion Cement: Baseline energy to failure \* 375



## Pre-stressed Cement Shearbond

Shearbond Test:

After Annular Seal test is complete, the force required to break the shearbond between cement and central pipe is measured.





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## Pre-Stressed Cement Shearbond

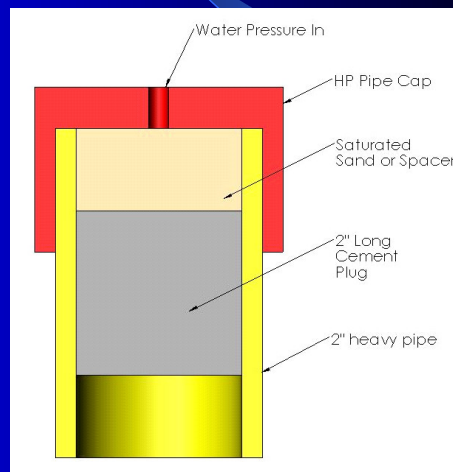
- Short pipe end mushroomed
- Approx 25,000 lbs applied force
- Shearbond >3,960 psi
- Pipe / cement bond did not break



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## Phase II Work Results – Hydraulic Bond

- Objective: Measure the ambient sealing properties of cement to water flow
- Results:
  - Baseline – 3,800 psi
  - Resin – 6,425 psi
  - Pre-stressed Cement – 6,000 psi



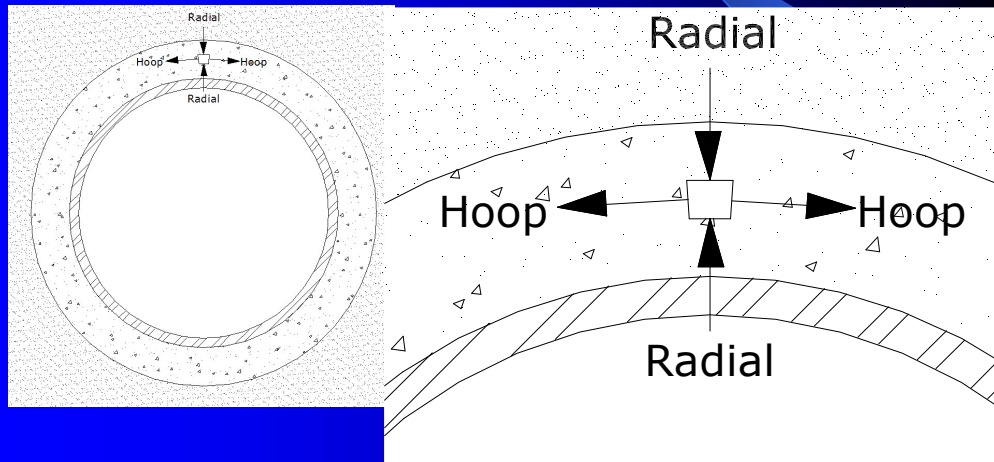




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## Phase II Work Results - Expansion

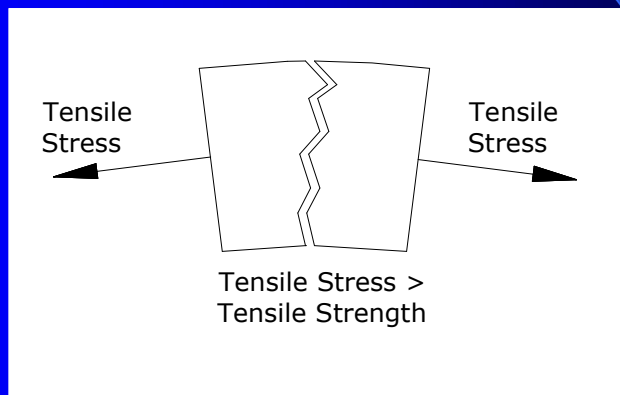
- Internal pipe pressure induces tensile (hoop) stress in the cement sheath.



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## Phase II Work Results - Expansion

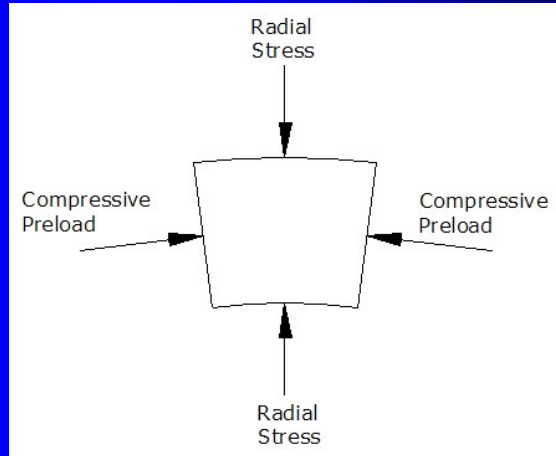
- Conventional non-expanding cements:
  - Cement fails in tension
  - When the induced tensile stress exceeds the cement tensile strength, failure (and a flow path) results





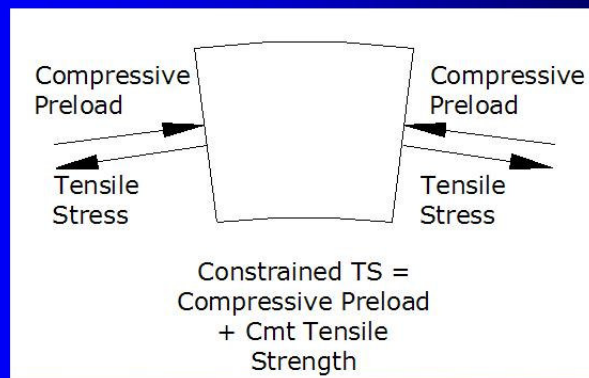
## Phase II Work Results - Expansion

- Controlled cement expansion creates a compressive preload



## Phase II Work Results - Expansion

- Compressive preload increases the cement tensile strength
- Constrained TS = compressive preload + unconstrained TS





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## Phase II Work Results - Expansion

- Measured pipe growth due to Pre-stressed cement cured at temperature and pressure
- Average pipe growth: 0.002”
- Induced compressive pre-stress: 2,600 psi
- Conclusion: Expanding cement effectively increases the nominal cement Tensile Strength by **2,600 psi**



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## Phase II and III Conclusions

- All Phase II and III Objectives are met
- Successful lab testing with Resin and Pre-stressed cement
  - Very high HTHP Annular Seal performance
  - Very high Shearbond
  - Very high Hydraulic Bond
  - Both Resin and Pre-stressed cement are controllable at HTHP conditions
- Successful field trials with Resin
  - 50+ successful plug jobs
  - One successful HTHP squeeze job
- Pre-stressed cement is ready for full-scale field testing

*Questions / Discussion*

## Questions and Answers

This section is a summary of the Q&A session at the conclusion of the IADC DeepTrek project presentation. Questions are listed and numbered; the answers follow in italics.

1. How would the Resin and Prestressed be placed in deep hot wells?

*The resin product requires special mixing equipment to blend the two liquid phases together. No solids are conveyed pneumatically into the equipment as is customary in conventional cement mixing equipment. Modest PPE is required to protect personnel from skin and eye contact with the product, and to prevent aspiration of fumes. Placement depends on the well situation, as the material has many unique properties. These include the ability to flow through standing well fluids without intermixing, or floating on top of well fluids as the situation dictates. The product generally does not have particles to screen out on formation faces. PRESTRESSED CEMENT mixing and placement is consistent with current Portland cement practices.*

2. How about using these products in very narrow annuli?

*Ultra Seal-R should be easier than conventional cements, due to the 100% fluid composition. Narrow annuli is an excellent application for both materials due to the superior tensile strength and ability to withstand stress.*

3. What is the friction of these slurries compared to conventional cements?

*Friction pressure is not appreciably different than conventional cements if weighting solids are present, and somewhat less if no solids are used. PRESTRESSED CEMENT has the potential to show less friction pressure than conventional cements because of the variety of particle sizes. Stability is good with no segregation.*

4. What is the application temperature of both products?

*Current applications have been performed with Ultra Seal-R up to 300 degrees F, and field compositions have been developed for PRESTRESSED CEMENT up to 500 degrees F. Work is continuing with Ultra Seal-R to raise the application temperature utilizing alternate base materials, extenders, and hardeners.*

5. How many jobs have been performed with these products?

*Ultra Seal-R has been used in over 100 field jobs, and the vast majority have been successful. A number of PRESTRESSED CEMENT field designs are complete and ready to go, but no jobs have been performed to date.*

6. What are the limitations of the systems compared to Portland cement?

*Limitations are volumes with Ultra Seal-R. Too much volume can make mixing difficult, as the current practices are to batch mix. There are no limitations with PRESTRESSED CEMENT.*

*Appendix G – Project Financial Summary*

Category	Phase I			Phase II			Phase III			Project Total		
	DOE	CSI	Total	DOE	CSI	Total	DOE	CSI	Total	DOE	CSI	Total
Personnel	\$333,259	\$272,851	\$606,110	\$226,623	\$302,445	\$ 529,068	\$145,340	\$ 331,733	\$477,073	\$ 705,222	\$ 907,030	\$ 1,612,252
Fringe	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -
Travel	\$ 8,280	\$ 5,711	\$ 13,991	\$ 6,562	\$ 21,298	\$ 27,859	\$ 9,310	\$ 13,982	\$ 23,292	\$ 24,152	\$ 40,990	\$ 65,142
Equipment	\$ 75,900	\$ 55,779	\$131,679	\$ 34,230	\$ 82,056	\$ 116,285	\$ -	\$ 21,775	\$ 21,775	\$ 110,130	\$ 159,610	\$ 269,740
Supplies	\$ 51,750	\$ (42,065)	\$ 9,685	\$ 38,549	\$ (4,662)	\$ 33,887	\$ 9,800	\$ (5,061)	\$ 4,739	\$ 100,099	\$ (51,788)	\$ 48,311
Outside Cost Share	\$ 48,300	\$ 3,988	\$ 52,288	\$200,129	\$234,714	\$ 434,843	\$191,100	\$ (182,850)	\$ 8,250	\$ 439,529	\$ 55,852	\$ 495,381
Consultants	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -
Other	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -	\$ -
Total Direct	\$517,489	\$296,263	\$813,752	\$506,093	\$635,850	\$ 1,141,943	\$355,550	\$ 179,580	\$535,130	\$ 1,379,132	\$ 1,111,694	\$ 2,490,826
G&A Indirect	\$ 54,056	\$ 22,505	\$ 76,561	\$ 26,874	\$ 22,345	\$ 49,219	\$ 22,013	\$ 19,938	\$ 41,951	\$ 102,943	\$ 64,788	\$ 167,731
Total Costs	\$ 890,313			\$ 1,191,162			\$ 577,081			\$ 2,658,557		
Awardee Cost Share	35.80%			55.26%			34.57%			44.25%		
DOE Share	64.20%			44.74%			65.43%			55.75%		
Awardee Total Cost	\$ 318,768			\$ 658,195			\$ 199,518			\$ 1,176,482		
DOE Cost	\$ 571,545			\$ 532,967			\$ 377,563			\$ 1,482,075		
Total Costs	\$ 890,313			\$ 1,191,162			\$ 577,081			\$ 2,658,557		

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