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SAND--82-7203 DE83 009393

# **Development of Modifications for Coflexip Flexible Drilling Pipe for High Temperature and Pressure Geothermal Service**

## **Final Report**

Gilbert J. Friese L'Garde, Inc. 1555 Placentia Avenue Newport Beach, CA 92663

Prepared by Sandia National Laboratories Albuquerque, New Mexico 87185 and Livermore, California 94550 for the United States Department of Energy under Contract DE-AC04-76DP00789

**Printed February 1983** 



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Printed in the United States of America Available from National Technical Information Service U.S. Department of Commerce 5285 Port Royal Road Springfield, VA 22161

NTIS price codes Printed copy: A04 Microfiche copy: A01

## DEVELOPMENT OF MODIFICATIONS FOR COFLEXIP FLEXIBLE DRILLING PIPE FOR HIGH-TEMPERATURE AND PRESSURE GEOTHERMAL SERVICE

### FINAL REPORT

December 1982

Gilbert J. Friese

L'Garde, Inc. 1555 Placentia Avenue Newport Beach, California 92663 (714)645-4880

Prepared For Sandia National Laboratories Under Contract Number 62-1247

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#### DEVELOPMENT OF MODIFICATIONS FOR COFLEXIP FLEXIBLE DRILLING PIPE FOR HIGH TEMPERATURE AND PRESSURE GEOTHERMAL SERVICE

#### Gilbert J. Friese Author

L'Garde, Inc. Newport Beach, California 92663

#### ABSTRACT

Coflexip (France) flexible drilling pipe can provide economies in drilling geothermal wells. However, the current liner materials cannot take the high temperatures (~250C) and pressures (~69 MPa).

Development was undertaken to replace the liner with higher temperature materials and, thus increase the temperature capability of the flexible pipe. DuPont Teflon PFA 350, L'Garde EPDM Y267 and L'Garde AFLAS 291 were considered but they all require backing by a closely woven stainless steel fabric to prevent extrusion.

A graphite-reinforced EPDM elastomer was developed which has the potential of meeting the pressure-temperature requirements without the metal fabric reinforcement.

#### FOREWORD

This program was sponsored by Sandia National Laboratories; Albuquerque, New Mexico, under Contract Number 62-1247. Billy Caskey (initially Don Wesenburg) was the Sandia Technical Program Manager who monitored program progress and suggested different technical approaches. Sandia's Charles Arnold, the materials specialist, researched many materials and recommended those that had the potential for meeting the requirements.

At L'Garde, Gilbert J. Friese was the principal investigator. Gordon Veal designed the simulation test equipment. Don Davis was responsible for fabricating the equipment and conducting the tests. Alan Hirasuna provided valuable suggestions in the areas of elastomer reinforcement and simulation testing.

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#### 1.0 INTRODUCTION AND SUMMARY

Flexible drilling pipe provides the potential for decreasing the cost of drilling geothermal wells by enabling reeling a continuous pipe rather than making and breaking joints of rigid pipe. The Coflexip flexible drilling pipe described in Section 2 uses a nylon liner which cannot survive the high temperatures. The objective of this program was to develop modifications such that the pipe can take 69 MPa pressures and 125C temperatures during drilling, and 2 MPa pressures and 250C temperatures during equipment down times.

The liner is backed-up by a flexible interlocking Zeta pipe which has gaps at the interlocks as large as one mm for 51 mm inner diameter pipe. If the liner is forced into these gaps by the pressure, flexibility if reduced and damage to the pipe will occur when a bending moment is applied.

Simple screening test equipment and more complex simulation test equipment were designed and built (Section 3) to evaluate candidate liner materials. Ten materials that had the potential for withstanding the environments were identified by Sandia and L'Garde. These were tested, and seven were rejected because they deleteriously softened at 250C or had inadequate elongation for the application (Section 4). The three materials which looked the best were Teflon PFA 350, L'Garde EPDM Y267 elastomer and L'Garde AFLAS 291 elastomer. However, they all suffered mechanical damage at 69 MPa pressure at 125C (Section 4).

Stainless steel fabric was successfully used to reinforce the downstream surface of the three materials by reducing the span the material must bridge (Section 5). The liner materials did not penetrate significantly into the metal fabric, and the metal fabric did not penetrate significantly into the Zeta pipe gaps.

In addition to the metal fabric reinforcement approach, a small development was undertaken in which EPDM and AFLAS elastomers were reinforced with Kevlar 29 and/or graphite chopped fibers in addition to Inconel 718 strands.

Also graphite-reinforced EPDM compounds were investigated and have a reasonable probability of meeting the requirements (Section 6). These compounds penetrated 1.0 and 1.5 mm gaps by 1 mm depth or less. Penetration of this amount is anticipated to be satisfactory because the material would be pushed out of the Zeta pipe gaps as the pipe was bent.

### 2.0 COFLEXIP FLEXIBLE DRILLING PIPE

Coflexip flexible pipe\* combines thermoplastic layers in a steel structure that has the flexibility of reinforced rubber hose and the strength and durability of steel pipe. It has advantages over conventional drilling pipe in that it can be reeled-up avoiding the time-consuming making and breaking of joints necessary with rigid pipe.

Dozens of different structures have been developed for specific applications, in sizes from 13 to 600 mm ID, and with working pressures up to 100 MPa. All of these structures, however, are derived from the same basic design (see Figure 1) which consists of three elements.

- Interlocking Z-section steel: This spiral-steel layer -- or carcass -- provides the strength for the high radial loads exerted by internal and external pressures, provides high fatigue strength, and keeps the pipe from kinking.
- o Armoring layers: These two layers are continuous, flat steel straps spiralling in opposite directions. Without detracting from flexibility, these layers add to the impact strength of the pipe and give it longitudinal tensile strength comparable to steel pipe.
- o Thermoplastic layers: Extruded thermoplastic layers inside and outside (construction varies for some applications) perform three important functions: the inner tube makes the pipe leakproof; both layers protect the steel components against corrosion; and they both offer good abrasion resistance.

The problem for geothermal applications is that the thermoplastic liner, which is generally polyamide 11 (nylon), has a melting point of about 250C. At lower temperatures, such as 125C, it is plastic and flows under high pressure. If the liner flows into the gaps of the interlocking Z-section layer (Zeta pipe), the pipe cannot bend and if forced, the Zeta pipe will be damaged.

#### \*Product of Coflexip; France



Figure 2 contains a photo of the Zeta pipe cross section, and a set of measured dimensions. The objective of this program was to develop a high temperature system which keeps the Zeta pipe gaps voided and thus the Zeta pipe functional. The conditions of interest are:

o 2 MPa pressure at 250C temperature

o 69 MPa pressure at 125C temperature

During drilling, the pipe will experience the high pressure, low temperature condition. If drilling stops, the low pressure, high-temperature condition will result.

Measurements of the Zeta pipe showed that it contracts and elongates a total of 9% or less. Therefore, the liner must have an ultimate elongation of more than 9%.

#### 2.1 DISCUSSIONS WITH COFLEXIP

In an effort to keep the development pertinent, discussions were held with Coflexip to assess constraints that manufacturing, handling, rig use, etc. may place on the design. A meeting was held with Coflexip and Services, Inc. to gain further information, as outlined below.

Q. What processes and machinery are used to make and apply the inner and outer liners?

A. Each layer of Coflexip pipe is put on in a separate operation. The nylon is extruded, cooled and placed on a reel. The Zeta pipe is fabricated over the nylon tube (which comes from a reel) and then rolled onto another reel. Middle and outer layers of nylon or rubber are extruded directly on the pipe and immediately cooled. Coflexip personnel didn't believe there would be problems with pressure or temperature limitations for other liner materials. The screw which feeds the material would probably be changed for a new material.

Q. Does Coflexip make a 150 mm ID, 69 MPa pipe that Sandia needs? If so, what are the different layers?

A. 150 mm ID, 69 MPa pipe has not been made. The configuration, from inside to outside, would probably be: a) high-temperature liner; b) Zeta pipe; c) flat-bound pressure armor and counterwound Zeta pipe; d) hightemperature liner; e) two layers of tensile armor; f) high-temperature rubber.





## Figure 2 Zeta Pipe Cross Section

Coflexip believed it was essential to have an outer layer of rubber to form a seal with the BOP. Tooling is available which compression molds rubber on the pipe in three meter lengths. They thought this machine would work economically with the L'Garde elastomers. Post cure equipment does not exist.

Q. Could a layer of axial flat steel ribbon (normally exterior to the Zeta pipe) be applied to the Zeta pipe interior instead?

Placing tensile armor on the inside is not a good idea since it has an uncontrolled gap. The Zeta pipe was invented solely to provide a minimum gap.

A. What liner materials, other than nylon, were evaluated during the development of Coflexip flexible pipe?

Perhaps 200 liners were evaluated. A list was not available.

Q. If a new high temperature liner material required specialized equipment that existed elsewhere, are any problems foreseen with receiving reels of liner for incorporating with the steel elements?

A. No problems.

#### 2.2 SYSTEMS ANALYSIS

The question was raised: Will an elastomeric liner slowly creep to the bottom of the well due to its own weight? Based upon the following simple analysis, this doesn't seem to be a problem.

If the liner receives no support from the Zeta pipe, the stress at the top of the liner is

 $s = \rho L$ 

where  $\rho$  is the material density and L if the liner length. If  $\rho = 11000 \text{Nm}^{-3}$  and L is 1500 m, the stress is  $16\frac{1}{2}$  MPa. Without support, then, the elastomer would elongate and break. (The ultimate stress and elongation of Y267 EPDM at room temperature are 14 MPa and 200%, respectively.)

What internal pressure, then, is needed to firmly hold the rubber to the Zeta pipe by friciton? This turns out to be

 $P = t_{\rho}/f$ 

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where t is the liner thickness (6 mm) and f is the steel-to-rubber friction coefficient (about one). The required pressure then is 0.07 MPa. This is considered negligible. For instance, atmospheric pressure increased by that amount only six meters down. And pressures greater than 2 MPa are expected.

With an internal pressure greater than 0.07 MPa, then, the elastomer in contact with the steel cannot move in any direction. Further away from this interface, the liner has to support its weight without creeping. The shear stress is

$$s_s = \rho t = 0.07 \text{ MPa}$$

EPDM 267 should not creep under such a load. Under another program, compressive creep was measured for 267 EPDM:

Compressive Stress (MPa)	Temp. (ºC)	Creep (m/m-yr)	Temp. (°C)	Creep (m/m-yr)
0.3	251	0.1	361	11
0.7	256	0.2	308	14
2.1	259	1.0	312	
		3		

•

#### 3.0 TEST EQUIPMENT

Simple test devices were designed and built to evaluate liner material and composite candidates. A more elaborate test was conceived, designed and built for testing the best concepts in a quasi dynamic simulation of actual operation.

#### 3.1 SCREENING TEST RIGS

Separate test fixtures were required and built for each of the test pressures: 2 and 69 MPa. The objective of these tests was to evaluate the extrusion resistance of the candidates at temperature.

The low pressure fixture is shown in Figure 3. It is placed in a heated hydraulic press and brought up to 250C temperature. A flat specimen is placed on top and the 2 MPa pressure is applied with the press. The gaps represent the gap in the Coflexip Zeta pipe. Fully expanded, the Zeta pipe has a gap of 1 mm. The distance that the material would penetrate these gaps is a parameter measured in this test. As it turned out, the pressure was so low that the material would not enter the gaps unless it became externally soft. Therefore, those materials which did not catastrophically soften at 250C survived this relatively benign evaluation.

For the high pressure evaluation, the Figure 4 fixture was developed. The cylinder provides lateral support for the material as the 69 MPa force supplied. This simulates the 3-dimensional conditions in the actual drill pipe. This figure is designed to work in conjunction with a heated hydraulic press which provides the force and the 125C temperature. A 38 mm diameter by 6 mm thick "hockey puck" of test material is the stand test specimen placed inside the device. The general procedure was as follows:

- 1. Apply 17.2 MPa pressure; measure and record penetration depth at 0, 5 and 10 minutes.
- 2. Repeat Step 1 for 34.5, 51.7, and 69 MPa.



Figure 3 Low Pressure Screening Test Fixture



### Figure 4 High Pressure Screening Test Fixture

#### 3.2 SIMULATION TEST RIG

The Coflexip Zeta pipe, when fully bent, has a maximum gap of 1 mm for 51 mm ID pips, respectively. The liner must bridge this gap while under temperature and pressure. In addition, during bending, the liner must elastically contract (or expand) about 9% for 51 mm ID pipe. During this process, there will be some abrasion between the liner and Zeta pipe.

Coflexip said that their nylon liner will weaken at 250C and enter the gap. This will result in the Zeta pipe breaking or being damaged when the pipe is bent. Overcoming this situation is a critical aspect to the development of geothermal Coflexip Drilling Pipe. Therefore, an evaluation test was developed which simulates these circumstances.

Section 3.1 covers Screening Tests which were used to narrow the field of candidates to a more economic number. The surviving candidates were then evaluated in the Simulation Test to a) evaluate their resistance to abrasion or other dynamic phenomena, and b) demonstrate if marginally extrusion resistant materials are satisfactory because they are squeezed out of the gap and do not remain to be pinched in the Zeta pipe.

The test section is shown in Figure 5. The material being tested as a liner is made into a hat-shaped specimen. Most of this hat bears against a piece of Coflexip Zeta pipe that is welded to the upper and lower steel pieces.

When Coflexip pipe is bent, the gaps on one side of the Zeta pipe narrow while the gaps on the other side broaden. For economy, in this simulation, there is no bending. The test section is elongated (or compressed) to open (or close) all gaps simultaneously. Similar to the Screening Tests, this fixture is designed to operate in conjunction with a heated 450,000 Newton hydraulic platen press. The press provides a convenient source for the force and the 250C temperature. The distance between the hydraulic press platens is regulated to provide the desired Zeta pipe gap.





The friction between the Zeta pipe and support tube is significant. If the tube had to take the full pressure, the static friciton coefficient must be

$$f_{s} \leq \frac{pA_{c}}{pA_{s}} = d/4l$$

**≤** 0.109

A high temperature, dry lubricant is needed and was used. The allowable coefficient of friction can be increased by a shorter Zeta pipe (per the above formula). Instead, the tube is being made slightly larger than the Zeta pipe so that the latter can be stressed to near yield. In this way the Zeta will carry at least half the pressure, and the friction coefficient can be as high as 0.2 or more.

The pressurization and instrumentation system is shown in Figure 6. Figure 7 contains photos of the completed system. This system pressurizes the test liner interior to any pressure up to 69 MPa. Because of this large pressure range, the accumulator, A, is used for pressures under 9 MPa and the pressure intensifier, I, is used for pressures between 9 and 69 MPa.

A detailed procedure for operating this system is provided in the Appendix. In summary, say a high pressure (>9 MPa) is desired. Valve V2 is closed to isolate the low pressure accumulator. The intensifier piston is brought fully down and held there. A vacuum is pulled at V1, and silicon oil is back-filled into the test section and intensifier.

Nitrogen pressure is applied to the bottom of the intensifier from a nitrogen bottle and the regulator, R. Because the area ratio between the bottom and the top of the intensifier is 10.6, a nitrogen pressure of 7 MPa results in an oil or test pressure of approximately 74 MPa.

The nitrogen and oil pressures are read on gages Gl and G3. Temperature is read via a thermocouple strapped to the test fixture support tube. Finally, the pressure switch is connected to a 7-day clock (not shown). If the liner breaks or leaks while the system is unattended, the clock will stop thereby providing time of failure.



A:	3,000 psi, 11 cu. in. piston accumulator
BD1:	12,000 psi burst disk
BD2:	1,500 psi burst disk
G1:	15,000 psi pressure gage
G2,G3:	1,500 psi pressure gage
I:	10,000 psi, 5 cu. in., 10.6:1 pressure intensifier
R:	2,000 psi reducing & relief regulator
S1:	15,000 psi pressure switch
S2:	1,500 psi pressure switch
V1,V2:	45,000 psi valve
V3-V6:	4,000 psi valve
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Figure 6 Pressure and Instrumentation System Schematic



The insulated test section is in a platen hydraulic press. Most of the time, the press will be fully open -- giving the liner maximum opportunity to creep into the Zeta pipe gaps. At predetermined times, the gap will be closed using the press. The intensifier or accumulator will back off to accept the fluid, and the regulator will relieve the system of excess pressure.

The system was checked out with an EPDM Y267 elastomeric liner, and it operated as desired. The simulation works as designed but with the originally unanticipated metal fabric there are anomallies as described in Section 5.

#### 4.0 SCREENING TESTS

Sandia and L'Garde researched the literature and the following stateof-the-art materials were determined to be candidates for geothermal Coflexip drilling pipe:

> DuPont PFA Teflon 350 General Electric Noryl ENG 265 ICI Americas PEEK ICI Americas Victrex L'Garde AFLAS 291 Elastomer L'Garde EPDM Y267 Elastomer Polyamide 11 (Nylon)\* Raychem Stilin DuPont Tefzel 280 Union Carbide Udel

These were first tested at 2 MPa pressure and 250C. The nylon\* 11 (which is the current Coflexip liner), Noryl ENG 265, Vitrex, and Udel catastrophically softened. Tefzel 280 became very soft; it has an advertised melting point of 270C and 250C which does not provide any margin for this application. These post test specimens (except for Vitrex) are shown in Figure 8.

Raychem Stilin withstood 250C, 2 MPa (and subsequently 125C, 69 MPa) without any difficulty. However, this material's yield point is about 70 MPa at 10% strain -- too high a modulus and too strong for use in a flexible pipe.

ICI Americas PEEK was rejected untested for the same reason. It has only a 6% strain at its yield point, while at least a 9% strain is needed prior to a material's elastic limit. PEEK is very stiff with an elastic modulus of 1700 MPa -- impractical for this application.

The PFA Teflon 350, AFLAS 291, and EPDM Y267 survived the temperature and penetrated the gaps by less than 0.2 mm (Figure 9). These plus the base-

\*Specimens supplied by Coflexip



Figure 8 Materials Rejected Because of Poor Performance at 250C



Figure 9 Potential Liner Materials After 250C and 2 MPa

line nylon 11 were then tested at pressures up to 69 MPa. The test results are provided in Tables I through IV.

What is acceptable? The Zeta pipe has a radius of approximately 1 mm at the gap. The liner could penetrate the gap as much as 1 mm when it is fully open (1 mm wide). Then when the gap closes, the liner is likely to be pushed out. With greater penetration, there is no component of force pushing the material out, and failure is likely. Failure is assumed to occur when the pipe loses bending ability. Therefore, it will be assumed that penetration of 1 mm or less into a 1 mm gap is acceptable.

From Tables I through IV, using the above criteria, it is seen that the candidate materials at 125C can take the following pressures:

Nylon 11	34	MPa
Teflon PFA 350	39	MPa
AFLAS 291	28	MPa
EPDM Y267	17	MPa

None meet the 69 MPa requirement. (Of course, Nylon 11 is too soft at 250C and therefore does not meet the 250C, 2 MPa requirement as the others do.)

Figure 10 shows two of the specimens after exposure to 69 MPa pressure and 125C temperature. The elastomers, both AFLAS 291 and EPDM Y267, fail in shear as the unsupported material enters the gap. It them fails in tension as the material is pulled from the test fixture. The Teflon flows into the gap, and can be removed intact. Neither result is acceptable.

These data lead to the conclusion that the compounds which can take the high temperature need some reinforcement to limit the extrusion at the high pressure.

Pressure	Gap Width (mm)		
Room Temperature	1.0	1.5	2.0
0. 34.5 MPa	0.2	0.2	0.8
68.9 MPa	0.2	0.2	0.8
125°C Temperature		e de la	
17.2 MPa	0.5	0.5	1.0
34.5 MPa	1.0	1.5	1.5
51.7 MPa	1.5	1.8	2.3
68.9 MPa	3.0	4.1	5.6
250 <sup>0</sup> C Temperature	and strain the	melts	

## TABLE I Nylon 11 Penetration Distance (mm) Into Gap

## TABLE II EPDM Y267 Penetration (mm) Into Gap

Pressure	Gap Width (mm)		
125 <sup>0</sup> C Temperature	1.0	1.5	2.0
17.2 MPa	1.0	1.5	2.0
34.5 MPa	2.0	3.6	4.1
51.7 MPa	6.1	7.1	7.6
68.9 MPa	8.9	Full	Full
250 <sup>0</sup> C Temperature			
17.2 MPa	1.5	4.1	4.6
34.5 MPa	Full	Full	Full

Pressure	Gap Width (mm)		
125°C Temperature	1.0	1.5	2.0
17.2 MPa	0.5	0.8	1.0
34.5 MPa	0.8	1.3	2.0
51.7 MPa	1.5	2.5	3.0
68.9 MPa	2.3	3.0	5.1
250°C Temperature			
17.2 MPa	1.5	1.5	3.0
34.5 MPa	3.0	4.6	6.1
51.7 MPa	4.1	6.1	Full

	TABLE III				
PFA	350 Teflon	Penetration	(mm) Into	Gap	

		TABLE IV			
AFLAS	291	Penetration	(mm)	Into	Gap

Pressure	Gap Width (mm)		
125 <sup>0</sup> C Temperature	1.0	1.5	2.0
17.2 MPa	0.5	0.8	1.0
34.5 MPa	1.3	2.0	3.6
51.7 MPa	4.6	7.1	Full
68.9 MPa	7.1	Full	Full
250 <sup>0</sup> C Temperature			
17.2 MPa	1.5	2.0	8.1
34.5 MPa	Full	Full	Full
51.7 MPa			2



Figure 10 Potential Liner Materials After 125C and 69 MPa



#### 5.0 METAL FABRIC REINFORCEMENT

The tests of Section 4 showed that only the Teflon and elastomers can withstand the temperature and were elastic enough for geothermal application. However, neither can withstand the high pressure.

A potential solution to this problem is to reinforce sealing liner materials with closely woven metal fabric. Metal fabric has long been used with rubber and Teflon to produce high pressure hoses (with burst strengths as high as the required 69 MPa). For drilling pipe the metal fabric need not be used for strength, but only to reduce the gap that the Teflon or elastomer must bridge. The Coflexip Zeta pipe and other armor are available to take the pressure.

The concept was tried in the Screening Test using the hockey puck specimen with L'Garde EPDM Y267. Figure 11 shows the results of two tests. The difference between the right and left tests was the orientation of the weave with respect to the Zeta pipe. One can see that the steel filaments entered the Zeta pipe gap when oriented as on the right; when oriented as on the left, there was only slightly visible penetration. As expected, the latter is better, and is also easier to implement. The rubber specimens shown above the fabric have imprinted on them the fabric pattern. The rubber penetrated less than one filament diameter. (The upper edge of the left specimen was not protected by fabric, and therefore it failed locally.)

A metal fabric was evaluated in the simulation test set-up even though the test system was not designed for it. The simulation test elongates the rubber axially rather than bending it. Metal fabric keeps a constant diameter when it is bent, but its diameter decreases (increases) when it is elongated (contracted) longitudinally. Under pressure, however, the fabric is prevented from changing diameter. Therefore, it acts like rigid steel and cannot flex.

Because of this, only a 38 mm wide hoop of fabric was used in the first test of a Teflon PFA 350 hat. The specimen was held at 250C and 2.1 MPa with the Coflexip gaps fully open for 70 hours. Then the hat was compressed



Figure 11 Metal Fabric Reinforcement of L'Garde EPDM Y267

such that the gaps fully closed. This compression was repeated once, and the test was considered complete. As expected at this low pressure, the Teflon did not enter the gaps sufficiently to cause any problem. Apparently, the friction between the fabric and the Teflon was high (preventing slippage during compression) because the Teflon buckled below the edge of the fabric. The test emphasized that the simulation test is beyond its extended capability when fabric is used as a reinforcement.

A test was then attempted at 125C and 69 MPa pressure using a 13 mm wide piece of fabric. The seal at the hat rim leaked at 62 MPa pressure so the test was terminated.

The test was repeated with new materials, and again it leaked at 62 MPa pressure -- this time through a pin hole in the Teflon. Compressing the Zeta pipe buckled the Teflon adjacent to the fabric. Then elongating the Zeta pipe stretched the thinnest Teflon the most. This resulted in a weakened thin area adjacent to the fabric where a pin hole failure occurred.

Figure 12 shows the hat specimens after the high pressure tests with the metal fabric removed from one of them. The simulation test did confirm what was observed in the "hockey puck" screening tests. In the area unsupported by fabric, the Teflon filled the Zeta pipe grooves. In the fabric-supported area, the Teflon did not penetrate the metal fabric nor the fabric into the Zeta pipe gaps. The metal fabric surface pattern is permanently impressed on the Teflon surface, but the peak-to-valley distance is only about 0.08 mm, less than half a filament diameter.

The test data lead to the conclusion, but are too limited to conclusively prove that a combination of stainless steel metal fabric and Teflon or EPDM will satisfactorily perform as a liner for geothermal drilling applications. Both simulation tests showed that the liner material does not significantly penetrate the fabric. The fabric should be able to be bent without changing diameter, and therefore will work in the Zeta pipe. However, this could not be proven within the scope of this effort where the test specimens were stretched and compressed rather than bent.



Figure 12 Teflon Hat-Shaped Specimens Which Experienced 125C and 62 MPa Pressure

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#### 6.0 REINFORCED ELASTOMER DEVELOPMENT

While a metal fabric reinforced seal material will work, it is probably not the most economical approach. Therefore, some limited development work was done with reinforced elastomers.

Four approaches were taken:

- 1. AFLAS elastomer and chopped Kevlar.
- 2. EPDM elastomer and Inconel 718 wire.
- 3. AFLAS elastomer and chopped graphite.\*
- 4. EPDM elastomer and chopped graphite.\*

The first was a failure. Compounds similar to L'Garde AFLAS 291 were attempted with chopped Kevlar -- which has high strength and modulus. Mixing was impossible; the result was a material that fell apart readily. It could not be molded.

Fine Inconel 718 wire was chopped to about 13 mm lengths and treated to enhance adhesion to EPDM. This was then mixed with an elastomer similar to EPDM Y267. The results of testing are shown in Table V. Compound B was fully loaded with Inconel and had about three times as much Inconel as Compound A. Both compounds performed poorly, similar to the basic Y267. It appears that the rubber-Inconel adhesion was not good.

The graphite-reinforced AFLAS compounds were difficult to mix, but two were molded. The results of testing are shown in Table VI. The graphite did not produce a significant improvement; really, it is worse because the material creeps. This probably could be corrected, however, AFLAS 291 is a difficult compound to work with, and reinforcing makes it more difficult. In addition these properties do not lend themselves to extrusion, the preferred manufacturing process for drilling pipe.

The majority of this development effort involved graphite-reinforced EPDM compounds. Table VII shows that improvements over L'Garde EPDM Y267 were immediately obvious. In general, the percentage of graphite increased as the

\*Hercules, Inc. Graphite Fiber Type AS/1800 (1/2" long)

Gap Width (mm)	Pressure (MPa)	EPDM Y267	Inconel-Reinfo A	orced Compound B
1.0	17	1.0	1.5	1.0
1.0	34	2.0	3.0	2.3
1.0	52	6.1	6.1	3.3
1.0	69	8.9	7.1	7.4
1.5	17		1.8	1.3
1.5	34	Full	4.1	2.5
1.5	52		7.6	5.1
1.5	69		8.6	8.4

TABLE V Penetration Depth (mm) of Inconel-Reinforced EPDM Compound (125°C Temperature)

TABLE VI Pentration Depth (mm) of Graphite-Reinforced AFLAS Compound (125<sup>o</sup>C Temperature)

Gap Width	Pressure	AFLAS	Reinforced-Compound*			
(mm)	(MPa)	291	Α	В		
1.0	17	0.5	0.3	0.3		
1.0	34	1.3	0.5	0.5		
1.0	52	4.6	3.8	0.3		
1.0	69	7.1	Full	Full		
1.5	17	0.8	0.00	0.3		
1.5	34	2.0	0.05	1.0		
1.5	52	7.1	5.8	4.3		
1.5	69	Full	Full	Full		

\*Compounds A and B creep; values given are for 10 minutes of pressure application.

.

Pressure	EPDM	Reinforced Compound											
(psi)	Y267	A	В	C	D	G	Н	$\mathbf{I}_{\mathrm{ext}}$	J*	K	L	M*	N*
Gap Width	= 1.0	nm:											
.17	1.0	1.3	0.5	0.5	0.5	0.8	0.8	0.5	0.4	0.5	1.0	0.8	0.9
34	2.0	2.5	1.0	1.3	0.8	1.8	1.0	1.0	0.6	1.0	2.0	1.1	1.8
52 ·	6.1	5.1	2.8	3.0	4.3	3.0	2.0	2.0	2.0	3.6	4.1	2.4	4.8
69	8.9	Full		8.9	Full	6.9	5.1	6.1	5.0	6.1	Full	4.4	Full
Gap Width = 1.5 mm:		nm :		,4 19 						्म ्री २ • • २			
17	1.5	2.0	0.8	0.5	0.5	1.0	0.8	0.5	0.5	0.5	1.0	0.8	0.8
34	Full	3.3	2.0	1.3	1.5	2.0	1.3	1.3	0.9	1.0	2.0	1.0	1.5
52		4.8	3.6	3.0	7.6	4.1	2.5	3.0	2.3	3.6	2.5	1.8	2.3
69		Full	7.6	7.9	Full	Full	6.4	Full	5.8	6.1	Full	3.8	6.0

TABLE VII Penetration Depth (mm) of Graphite-Reinforced EPDM Compounds (125<sup>0</sup>C Temperature)

\*Test results for Compounds J, M, and N are the average results from two tests each.

compounding effort progressed. In addition, other modifications to the basic L'Garde EPDM Y267 were also made. The mechanical properties of some of these compounds are provided in Table VIII. Reinforcement increased the ultimate strength while decreasing elongation, a generally coupled phenomena which is acceptable for this application. For drilling pipe, the strain can be as low as 15% at the proportional limit and the higher modulus helps the material to bridge the gap.

Table IX contains the final set of compounds and the ones that have a high probability of meeting the requirements. Compound S is especially appealing. It penetrated both the 1.0 and 1.5 mm gaps by only 1 mm or less. In general, the shorter cure time was also beneficial, the compounds of Table VII were each cured for an hour. Tests were also run at 250C and 2.1 MPa. There was no indication that the high temperature was a problem. Also, the compounds did not penetrate the gaps at all.

Table X provides physical properties. The ultimate elongations are low (40-60%), and these are probably overstated by our method of measurement. By ASTM methods, the values could be as much as a third less. However, the compounds should meet the self-imposed requirement for 15% elongation at the elastic limit.

The limited compound development effort indicates that graphitefilled EPDM has potential for the difficult flexible drilling pipe requirements for geothermal drilling. Section 7 provides recommendations for future work.

	EPDM				Reinfo	rced Co	mpound			
Property	Y267	С	G	Н	O	J	K	L	М	N
Specific Gravity	1.12	1.15	1.14	1.17	1.17	1.17	1.19	1.19	1.19	1.20
Ultimate Strength (MPa)	12	22	23	25	20	23	25	26	29	27
Ultimate Elongation (%)	200	50	60	60	80	80	85	45	60	75
"Ultimate" Modulus (MPa)	6	44	39	42	24	27	29	56	50	35
Hardness	92	93	95	95	95	94	95	96	95	95

TABLE VIII Mechanical Properties of Graphite-Reinforced EPDM Elastomers

↓ 3 2.

Gap Width	Pressure	EPDM	Graphite-Reinforced Compound*						
(mm)	(MPa)	Y267	0	<u>Р</u>	QQ	R	S		
1.0	17	1.0	0.5-0.3	0.5-0.0	0.3-0.0	0.5-0.3	0.3-0.3		
1.0	34	2.0	0.5-0.5	0.5-0.3	0.5-0.5	0.5-0.5	0.3-0.5		
1.0	52	6.1	0.5-0.8	0.5-0.5	0.5-0.5	0.5-0.5	0.5-0.5		
1.0	69	8.9	1.5-2.0	1.5-0.8	1.0-1.0	1.3-0.8	1.0-0.5		
1.5	17	1.5	0.5-0.3	0.3-0.0	0.3-0.0	0.0.0.3	0.3-0.3		
1.5	34	Full	0.5-0.5	0.5-0.3	0.5-0.3	0.3-0.5	0.3-0.5		
1.5	52	<b></b>	0.8-1.5	0.5-0.5	1.0-0.3	0.8-0.8	0.5-0.8		
1.5	69		2.0-2.3	1.5-1.0	3.0-1.0	1.0-1.0	1.0-1.0		

TABLE IX Penetration Depth (mm) of Final Graphite-Reinforced EPDM Compounds (125<sup>0</sup>C Temperature)

\*Two values are given for each compound. The left value is for a 60 minute cure, and the right value is for a 20 minute cure.

Property	EPDM Y267	Graphite-Reinforced Compound*							
Specific Gravity	1.12	1.24	1.26	1.26	1.28	1.29			
Ultimate Strength (MPa)	12	27 25 30	26 29 26	29 30 23	30 26 26	30 32 26			
Ultimate Elongation (%)	200	50 40 45	45 55 50	50 60 50	40 40 50	45 60 60			
"Ultimate Modulus" (MPa)	6	54 62 68	56 52 50	61 51 45	72 66 54	70 54 44			
Hardness	92	95	95½	95	95 <sup>1</sup> 2	95½			

TABLE X Mechanical Properties of the Final Graphite-Reinforced EPDM Compounds

\*If three values are given under a given compound for a particular property, they are, in descending order, for a 60 minute, 30 minute and 20 minute cure.

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#### 7.0 CONCLUSIONS AND RECOMMENDATION

The results of the Development of Modification For Coflexip Drilling Pipe For High Temperature and Pressure Geothermal Service indicate that:

- Teflon PFA 350, EPDM Y267 and AFLAS 291 will meet the pressuretemperature requirements if they are backed by closely woven stainless steel metal fabric, and
- Graphite-reinforced EPDM elastomer has the potential of meeting the pressure-temperature requirements without the fabric back-up.

Based on the development data L'Garde believes that metal fabric reinforcement can be bent without changing its diameter. However, such a test could not be conducted within the scope of the program; therefore, this uncertainty exists regarding Item 1.

More work is required if a metal fabric tube is to be avoided by using the simpler reinforced elastomer concept. It is recommended that the following effort be accomplished:

- 1. Do additional compound optimization. The data show consistent results at 54 MPa pressure and promising results at 69 MPa.
- Run stress-strain curves to determine if the compounds are sufficiently elastic (~15% strain before the elastic limit).
- 3. Test the best compound(s) in the Zeta pipe simulation test setup.
- 4. Determine if the best compound(s) are extrudable.

APPENDIX COFLEXIP TEST PROCEDURE



APPLI	CATION		REVISIONS									
NEXT ASSY	USED ON	REV.	DESCRIPTION		DATE	APPROVE						
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TP10474 Sheet 2 of 7

#### COFLEXIP TEST PROCEDURE

#### 1.0 TEST SET-UP

- 1.1 Expand Zeta pipe approximately ‡" so that hot section rim is entering its mating part. <u>Hydraulic force must be limited to 4 tons</u>; if there is no measuring equipment, stop applying force when it becomes noticeably harder, or the part has increased 3/8 inch in length -- whichever occurs first.
- 1.2 Bolt on top section evenly. When the gap between the top and its mating part goes to near zero, remove from expansion device.

1.3 Torque all  $\frac{1}{2}$ " bolts to 400 inch-pounds gradually and evenly.

1.4 Place test section and oil pan (but no shim material) in the center of the hydraulic press platen, and hook up the oil line.

- 1.5 Hook up  $N_2$  bottle that has at least a pressure of the test pressure plus 800 psi for a low pressure test and at least 10% of the test pressure plus 800 psi  $N_2$  for a high pressure test. Leave  $N_2$  bottle valve closed.
- 1.6 Open all valves on test panel except V5; close V5.
- 1.7 Hook vacuum pump to V4 and evacuate gas line. When the vacuum is greater than 29 inches Hg, close V4 and V3, and remove pump.
- 1.8 Hook vacuum pump (with glass accumulator in the line) to V1 and evacuate to greater than 29 inches Hg. Close V1 and remove pump.
- 1.9 Place approximately 1000 cc of silicon oil in a bag, and attach to V1. Make sure that the plastic hose is full of fluid before attaching.

1.10 Open V1 and allow fluid to drain into system.

- 1.11 Close V1 and open V4. Leave low pressure, unnumbered valve next to V1 open. Place bag (still hooked up to V1) on floor.
- 1.12 Vent hydraulic press so that it is fully open, and confirm that it is. Turn regulator fully counterclockwise (OFF). Open N, bottle valve.

- 1.13 Using regulator, apply 200  $\pm$  20 psi to G2 (and G1).
- 1.14 Measure and record the test section gap in the front and back.
- 1.15 Measure and record the largest and smallest gaps between the test section and upper hydraulic press platen. Average these two readings and record the resulting valve.
- 1.16 Relieve pressure by rotating regulator fully counterclockwise.
- 1.17 Pump the hydraulic press until its pressure gage stabilizes at 300±30 psi (approximately 5 tons).
- 1.18 Measure and record test section gap in front and back.
- 1.19 Vent hydraulic press to zero load. So de terme a serie de la serie de la
- 1.20 Place sheet or plate metal (steel preferred) under the oil pan that has a thickness no less than the average recorded in 1.15 and no greater than 0.032 inch more.

- 1.21 Using regulator, apply 200 psi to G2. Check that the press is fully open and that the test section bears against the top platen.
- to baseling the second graph pro-1.22 Hook up thermocouple to test section.

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1.23 Insulate the test section.

#### 2.0 TESTING

- 2.1 Heat-Up and Pressure Control. Start all of the steps under 2.1 in the order given; however, some steps may not be finished before the next step is started. That is, read ahead.
  - 2.1.1 Turn on platen heaters, and continually adjust to 40F above the test temperature.
  - 2.1.2 Take readings of all temperature and pressure gages about every 15 minutes and at every event.
  - 2.1.3 When G2 becomes greater than the test pressure or 600 psi (whichever is less), turn regulator counterclockwise until G2 is 100±50 psi. If G3 goes to 0 psi and G2 is still greater than the test pressure or 600 psi (whichever is less), vent some oil by cracking V1 until the G2 pressure drops to 100±50 psi. Repeat this latter step anytime the G2 or G3 pressure exceeds the lesser of the test pressure or 600 psi (and G3 is 0 psi).
  - 2.1.4 When the thermocouple reaches 10F less than the test temperature, adjust the platen temperature (initially downwards) until the thermocouple stabilizes at the test temperature ±5F.
- 2.2 Continue to adjust temperature per 2.14, and read and record temperatures and pressures per 2.1.2 as the following steps are conducted.
- 2.3 Turn the regulator full counterclockwise until G3 reads 0 psi (if not already done).
- 2.4 If G2 is less than 300 psi, pump hydraulic press until <u>either</u> the pump press gage or G2 reads 300 psi.
- 2.5 <u>If and only if</u> G2 is 300 psi and the press pressure is less than 300 psi: Close V2 and then open V1; pump hydraulic press until its gage stabilizes at 300±30 psi on the press pressure gage; close V1 and open V2.

2.6 Remove insulation.

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- 2.7 Measure and record test section front gap.
- 2.8 Apply 200±20 psi to G3 by using the regulator.
- 2.9 Vent the hydraulic press pressure. Measure the test front section gap.

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- 2.10 Insulate the test section. The section and held a section section section and
- 2.11 <u>HIGH PRESSURE (1100-10,000 psi) TEST ONLY</u>: Close V2 and V4, and then open V3. (G1 will go to about 2000 psi.)
- 2.12 Slowly change the G1 or G2 reading to the test pressure  $\pm 5\%$ . If the pressure overshoots, back off the regulator to less than the test pressure and try again. Read and record all pressures and temperatures.

This is the start of the test.

- 2.13 Set the timer. Verify that it is running.
- 2.14 Occasionally, adjust temperatures and pressures if out of spec. Read and record all pressures and temperatures.
- 3.1 Seventy (70) hours after start of test (Step 2.12), record all temperatures and pressures.
- 3.2 Remove the insulation. Measure the test section front gap.
- 3.3 Slowly rotate the regulator counterclockwise until gas starts to vent, then rotate clockwise slightly.
- 3.4 Pump the hydraulic press until <u>its</u> gage stabilizes at 10% of the test pressure. Read and record all temperatures, pressures, and the test section front gap.

- 3.5 Stabilize the press pressure at the next value divisible by 100. (For instance, if the test pressure is 300 psi, increase from 30 psi to 100 psi; if the test pressure is 10,000 psi, increase from 1000 psi to 1100 psi.) Read and record temperatures, pressures, and the test section front gap.
- 3.6 Stabilize the press pressure at a value 100 psi higher. Read and record all temperatures, pressures and the test section front gap. Continue doing this until the press pressure is 1500 psi greater than 10% of the test pressure, or until the gap measurements are within 0.02 inch of that measured at the start of the test.
- 3.7 Vent the hydraulic press to zero load.
- 3.8 Increase the regulator pressure until the test pressure is reached. Slowly back off the regulator until gas starts to escape, then rotate it slightly clockwise to stop gas loss.
- 3.9 Repeat steps 3.5 through 3.7.
- 3.10 Rotate regulator fully counterclockwise (P3 of zero).
- 3.11 Open V1 and then V2.
- 3.12 Pump hydraulic press to about 200 psi on its gage.
- 3.13 Vent hydraulic press.
- 3.14 Disconnect test section.

3.15 Disassemble.

3.16 Remove carefully. Push on bottom of hat rather than pulling on top for most of removal operation.

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## COFLEXIP TEST DATA SHEET

Date \_\_\_\_\_ Test Pressure \_\_\_\_\_ Test Temperature \_\_\_\_\_

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	Temp	erature ('	°F)	Pres	sure (	psig)	Gap (	inch)			
Time	Test Section	Upper Platen	Lower Platen	G1	G2	G3	Front	Back	Step No.	Press Pres.	Comment
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A: 3,000 psi, 11 cu. in. piston accumulator

BD1: 12,000 psi burst disk

BD2: 1,500 psi burst disk

Gl: 15,000 psi pressure gage

- G2,G3: 1,500 psi pressure gage
- I: 10,000 psi, 5 cu. in., 10.6:1 pressure intensifier

R: 2,000 psi reducing & relief regulator

- S1: 15,000 psi pressure switch
- S2: 1,500 psi pressure switch
- V1,V2: 45,000 psi valve
- V3-V6: 4,000 psi valve

Pressure and Instrumentation System Schematic

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