Energy

**G
E
O
T
H
E
R
M
A
L**

890/5WB. (3)
6-17

DR-1090-1

DOE/SF/11537-1
(DE85003232)

**GEOHERMAL ELASTOMERIC MATERIALS TECHNOLOGY
APPLICATION (GEM-TA) PROGRAM**

Final Report for the Period August 1981—August 1983

By
A. R. Hirasuna
D. L. Davis
G. J. Friese
J. W. Trailer

MASTED

August 1984

Work Performed Under Contract No. AC03-81SF11537

**L'Garde, Inc.
Newport Beach, California**

**Technical Information Center
Office of Scientific and Technical Information
United States Department of Energy**

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

This report has been reproduced directly from the best available copy.

Available from the National Technical Information Service, U. S. Department of Commerce, Springfield, Virginia 22161.

Price: Printed Copy A07
Microfiche A01

Codes are used for pricing all publications. The code is determined by the number of pages in the publication. Information pertaining to the pricing codes can be found in the current issues of the following publications, which are generally available in most libraries: *Energy Research Abstracts (ERA)*; *Government Reports Announcements and Index (GRA and I)*; *Scientific and Technical Abstract Reports (STAR)*; and publication NTIS-PR-360 available from NTIS at the above address.

GEOTHERMAL ELASTOMERIC MATERIALS
TECHNOLOGY APPLICATION (GEM-TA) PROGRAM
FINAL REPORT
AUGUST 1981 - AUGUST 1983

LTR-84-AH-106
August 1984

A.R. HIRASUNA
D.L. DAVIS
G.J. FRIESE
J.W. TRAILER

L'GARDE, INC.
1555 PLACENTIA AVENUE
NEWPORT BEACH, CA 92663

PREPARED FOR
UNITED STATES DEPARTMENT OF ENERGY
SAN FRANCISCO OPERATIONS
FOSSIL GEOTHERMAL AND SOLAR DIVISION

CONTRACT NO. DE-AC03-81SF11537

FORWARD

The technical information reported herein was developed by L'Garde, Inc., Newport Beach, California under the Geothermal Elastomeric Materials Technology Application (GEM-TA) Program, DOE Contract No. DE-AC03-81SF11537.

The work was sponsored by DOE/San Francisco Operations, Fossil Geothermal and Solar Division. The program was managed for DOE by Messrs. Anthony Adduci, Gerald Katz, and Wayne Bryan. Messrs. Clifford McFarland, David Allen, and Dr. Robert Reeber were the cognizant individuals at DOE Headquarters.

The effort was directed for L'Garde by Mr. Alan Hirasuna. The development of this report and the technology advancement achieved under the GEM-TA program are also attributable to the following personnel: Mr. Gilbert Friese (Experiment Planning, Evaluation, and Coordination), Messrs. Don Davis and James Trailer (Fabrication and Evaluation Testing). This report was meticulously prepared by Ms. Patricia Wall.

Invaluable information and support which was vital, because GEM-TA was comprised of several small quick efforts, was provided by personnel from Government laboratories and industry. The following were especially helpful: Mr. Gerry Chess (Peabody Floway), Messrs. Thomas Hinrichs and David Pierce (Imperial Magma), Messrs. Al Stetson, David Huey, and George Hosang (Solar Turbines) on the Lineshaft Pump Bearing Task; Messrs. Barna Toekes (Sperry Research), Jerry Barsoumain (Parker Seal), Andy Mitchell and Jack Leonhart (DuPont), John Krynowski (Goodrich), Alan Sohlo (3M), Jim Goodson (Regal) on the Gravity Head System Compatibility Task; Mr. Bert Dennis (LANL) on the Electrical Elastomers Task; Drs. Bob Hanold (LANL) and Chris Allen (Union Oil) on the Explosive Stimulation Task; and Mr. Roland Pettitt and Dr. John Rowley (LANL), Messrs. Gilbert Lamberson and Richard Jetter (Dowell) on the Cementing Wiper Plug Task.

STATEMENT OF OBJECTIVES

Apply benefits of successful Geothermal Elastomeric Materials developments by DOE/L'Garde to elastomers problems being experienced on various geothermal projects.

ABSTRACT

In 1979, L'Garde, Inc. completed the development of several geothermal elastomer compounds for the U.S. Department of Energy (DOE) Division of Geothermal Energy under Contract DE-AC03-77ET28309. Major advances in the state of the art were achieved with successes at extreme conditions such as temperatures up to and exceeding 320C (608F), pressures up to and exceeding 138 MPa (20,000 psi), and fluids ranging from brines to hydrocarbons.

Because various geothermal projects had many elastomers problems and their solutions were critical to the project success, the DOE sponsored this effort to help provide the advantages of the earlier developed technology described above. This Final Report is a comprehensive description of the work performed by L'Garde, Inc. on the Geothermal Elastomeric Materials Technology Application (GEM-TA) Program under DOE/San Francisco Operation, Fossil Geothermal and Solar Division Contract DE-AC03-81SF11537. The technology applications supported are as follows: pump lineshaft bearings; seals for Freon 114[®], synthetic hydrocarbon, and brine service; electrically insulative seals for logging tools; seals for nitrate salt explosive and steam service; and cementing wiper plugs. In addition there were minor efforts to further disseminate information associated with the elastomer development and case history experiences.

TABLE OF CONTENTS

1.0	INTRODUCTION AND SUMMARY	1
1.1	Introduction	1
1.2	Summary	3
1.2.1	Pump Lineshaft Bearings (Section 2.0)	3
1.2.2	Elastomer Compatibility Tests For Sperry System (Section 3.0)	4
1.2.3	Electrical Elastomer Compounds (Section 4.0)	5
1.2.4	Explosive Stimulation Experiment Seals (Section 5.0)	6
1.2.5	Brief Minisupport Tasks (Section 6.0)	7
2.0	LINESHAFT PUMP BEARINGS	11
2.1	Pump Bearing Background	11
2.2	Rubber Lineshaft Bearings	11
2.3	Test Specimens	13
2.4	Test Conditions	14
2.5	Test Results	14
2.6	Conclusions Regarding Elastomeric Lineshaft Bearings	18
3.0	ELASTOMER COMPATIBILITY FOR SPERRY GRAVITY-HEAD GEOTHERMAL ENERGY CONVERSION SYSTEM	21
3.1	Summary	21
3.2	Elastomer Compound Selection	24
3.3	Test Setup	27
3.4	Test Procedure	31
3.5	O-Ring Temperatures	35
3.6	Pressure	40
3.7	Test Results and Evaluation	41
3.7.1	Mobil SHC224/234 Oil	41
3.7.2	Freon 114®	45
3.8	Recommendations	48
4.0	ELECTRICAL COMPOUNDS FOR LOGGING TOOLS	53
4.1	Background	53
4.2	Development Approach	55
4.3	Development	58
4.3.1	Formulation Effort	58
4.3.2	Experimental Results	60
4.4	Conclusions Regarding Insulative Compounds	64
5.0	GEYSERS EXPLOSIVE STIMULATION EXPERIMENT SEALS	67
5.1	Background	67
5.2	Need For Y267 EPDM	67
5.3	Y267 EPDM Seals Provided	69
5.4	Experiment Results	69

TABLE OF CONTENTS (Concluded)

6.0	BRIEF MINISUPPORT TASKS	71
6.1	Operator Briefings	71
6.2	Cementing Wiper Plug	73
6.3	Spontaneous Technology Transfer	74
6.4	Presentation of Papers at Technical Symposia	76
7.0	REFERENCES	77
APPENDICES:	A. DC Volume Resistivity of Insulating Materials (ASTM D257-78)	
	B. Geothermal Packers and Packer Elastomers	
	C. High-Temperature Y267 EPDM Elastomer Field and Laboratory Experiences, August 1981	
	D. A Proven Elastomer Compound for Extremely Hostile Geothermal and Oilfield Environments	

LIST OF FIGURES

Figure 1-1.	Post Mortem Otis Packer With Y267 EPDM Seal After Geothermal Hydraulic Fracture At Union Well, Baca, NM. Seal is Like New.	2
2-1.	Test Bearing Configuration, Dimensions in mm and (Inches).	13
2-2.	Y267-CXIX-2 and 317-II-2 Bearings In As-New Condition After Testing At 93C (200F) for 100 Hours.	16
2-3.	Y267-CXIX-3 Bearing Showing Chunking After Testing At 182C (360F) for 50 Hours.	16
2-4.	317-II-4 Bearing Showing Well Controlled Abrasion After Testing At 182C (360F) For 50 Hours.	16
3-1.	Parker EPDM E962-85 Exposed to Mobil SHC224/234 Oil at 10.3 MPa ΔP for 140 Hours.	23
3-2.	3M Fluorel Cpd C [®] Exposed to Mobil SHC224/234 Oil at 10.3 MPa ΔP For 140 Hours.	23
3-3.	Parker Fluoroelastomer V747-75 Exposed to Freon 114 [®] at 10.3 MPa ΔP For 140 Hours.	25
3-4.	Goodrich EP EPCAR [®] Exposed to Freon 114 [®] at 10.3 MPa ΔP for 140 Hours.	25
3-5.	O-Ring Test Piping and Instrumentation Diagram.	28
3-6.	Test Section.	30
3-7.	As-Measured 2-329 O-Ring Groove Dimensions In Inches	29
3-8.	O-Ring Test Facility.	32
3-9.	Test Section Area, Pressure Amplifier and Back Pressure Accumulator.	32
3-10.	Test Section.	33
3-11.	Temperature Controls and Strip Chart Recorder.	33
3-12.	Front Face Pressure Accumulators.	34
3-13.	Mobil Oil Test T6 Temperature History.	36
3-14.	Freon 114 [®] Test T6 Temperature History.	37
3-15.	Mobil Oil Test Temperature Gradients.	38

LIST OF FIGURES (Concluded)

Figure 3-16.	Freon 114 [®] Test Temperature Gradients.	39
3-17.	Mobil Oil Test Pressures Histories	42
3-18.	Freon 114 [®] Test Pressure Histories	43
3-19.	O-Rings After Testing With Mobil SHC224/234 Oil.	46
3-20.	O-Rings After Testing With Freon 114 [®]	49
4-1.	Scale of Specific Resistance of Various Materials, "Engineering Uses of Rubber," edited by A. T. McPherson and Alexander Klemm, p. 280, Reinhold Publishing Corpor- ation. 1956.	54
4-2.	Schematic of Major Types of Resistivity-Temperature Curves.	55
4-3.	Current Through Y267 EPDM at Temperature.	56
6-1.	Dowell Y267 EPDM Cementing Wiper Plug After 280C (536F) Exposure. Note As-New Condition.	74

LIST OF TABLES

Table 2-I.	Summary of Dynamic Tests at Solar Turbines	15
2-II.	Summary of Static Immersion Tests at Solar Turbines	17
2-III.	Comparison of Static Immersion Tests on ECT Program	17
3-I.	Elastomer Test Compounds	21
3-II.	Test Conditions	22
3-III.	Elastomer Properties	27
3-IV.	Time Averaged Temperatures	35
3-V.	O-Ring Reconstructed Average Temperatures	40
3-VI.	Mobil SHC225/234 Oil Test Observations	44
3-VII.	Freon 114 [®] Test Observations	45
4-I.	Characterization Properties	57
4-II.	Geothermal Electrical EPDM Candidate Formulations	59
4-III.	Compound Characteristics	62
4-IV.	Resistivities at Temperature and Other Conditions	63
5-I.	Results of Immersion Tests in Explosive	68
6-I.	Organizations Briefed	72
6-II.	GEM-TA Papers	76

1.0 INTRODUCTION AND SUMMARY

The introduction describes the background which provided the basis for this program, the Geothermal Elastomeric Materials Technology Application (GEM-TA) Program. The summary provides an executive synopsis of the accomplishments achieved over the course of this contract.

1.1 Introduction

In 1979, L'Garde, Inc. completed a contract for the Department of Energy (DOE), entitled the Geothermal Elastomeric Materials (GEM) Program¹, the objective of which was to develop geothermal elastomer compounds. Prior to the conclusion of that work elastomers which performed reliably at geothermal temperatures of the order of 260C (500F) or higher did not exist.

Under the aegis of the GEM Program EPDM, EPDM/Viton®, Viton®, and AFLAS® compounds were developed which exceeded the requirement to perform as a casing packer seal at 260C (500F) for 24 hours. However, one compound, Y267 EPDM, was eminently successful in various applications up to temperatures in excess of 316C (600F) and pressures in excess of 138 MPa (20,000 psi). The successes were experienced both in the laboratory and in actual field applications. Figure 1-1 shows an Otis Engineering Corp. packer with a Y267 EPDM element after operating successfully in a hydraulic stimulation experiment sponsored by DOE/Los Alamos National Laboratory (LANL) and implemented by Republic Geothermal in a Union Geothermal well at the Baca, New Mexico. When retrieved, the seal element was "as new" as contrasted to then existing technology which would have experienced severe degradation of the seal and probable failure under the same circumstances. Reference 2 summarizes 17 laboratory and 32 field case histories where Y267 EPDM yielded superior successful performance.

Because various geothermal projects had many elastomers problems and their solutions were critical to the project success, the DOE sponsored the GEM-TA Program to help provide the advantages of the successful elastomer developments of the GEM Program to geothermal problem applications. This application of DOE technologic success to solve intractable geothermal problems completes the technology development cycle

Figure 1.1. Post-Mortem Otis Packer With Y267 EPDM Seal After Geothermal Hydraulic Fracture At Union Well, Baca, NM. Seal is Like New.

from initial investment to end-use pay-off. It also promotes more extensive use of the technology which implies more extensive benefit derived from the investment.

1.2 SUMMARY

The GEM-TA Program was a two-year program initiated in August 1981. The objective of the program was to provide the advantages of a highly successful DOE development for the benefit of other DOE geothermal projects. Specific problem elastomer applications which were critical to the development progress of other individual projects were selected for application of the GEM technology. Each comprised a miniproject and are summarized.

1.2.1 Pump Lineshaft Bearings (Section 2.0)

A significant segment of the geothermal resource requires downhole feed pumps which push the geothermal fluid out of the well. One type of pump which is a viable candidate is the lineshaft pump. It has an electric motor at the wellhead safe from the pervasive fluids and a centrifugal pump downhole driven by a driveshaft or lineshaft connecting the motor and pump. This configuration using elastomeric lineshaft bearings is a classic one and has been very successfully used for water pumps for decades. Elastomeric lineshaft bearings are unsurpassed for situations where the bearing is exposed to low viscosity fluids and especially with particulates such as water with sand. It provides a simple system where the pumped fluid flows by and through the exposed bearing providing the bearing lubrication. There is no need for enclosures to protect the bearings nor for special lubrication systems. This simple bearing configuration has proven itself over and over for two applications -- lineshaft water pumps and boat/ship propeller shafts. However as anticipated, at geothermal temperatures the elastomeric bearings become unreliable. Other types of bearings are candidates but the inherent advantages of the elastomeric bearings are especially applicable to the geothermal application making it most desirable for several reasons if elastomers can be made to work. Given the existence of an excellent geothermal elastomer, Y267 EPDM, a natural potential marriage exists between it and the lineshaft bearing application.

In 1981, Solar Turbines in San Diego was researching the lineshaft bearing problem for Brookhaven National Laboratory (BNL)\DOE. They had developed a bearing test rig to evaluate various types of bearing

materials. It rotated a 25.4 mm (1 inch) shaft in hot brine and applied a side load to the test bearing which was a constant 445N (100 lbs.) for these tests. They were highly aware of the elastomeric bearing technology and considered it to be a good candidate for this intractable application.

A small effort was accomplished under the GEM-TA Program which included the following:

- Research elastomeric bearing requirements (L'Garde)
- Develop one elastomer compound (L'Garde)
- Test bearings made of the new compound and Y267 EPDM (Solar)
- Evaluate results (Solar, L'Garde)

Only a small effort was possible given the budget constraints. The objective was to hopefully determine if elastomeric bearings were a possible candidate at 150-200C (302-392F). Looking at only one new compound was a high-risk approach necessitated by the limited budget, and would yield no information if the test results were negative.

Fortunately, the one new compound (317) yielded improvements over the Y267 EPDM. At 93C (199F) both performed flawlessly in the 100 hour test. In the 50 hour, 182C (360F) tests, both suffered some damage. The Y267 EPDM bearing appears to have lost material through chunking. The 317 bearing lost less material and through a well controlled abrasion.

This brief evaluation indicates that rubber bearings may be possible at geothermal temperatures. Pursuit of this possibility warrants a bonafide elastomer compound development effort.

1.2.2 Elastomer Compatibility Tests For Sperry System (Section 3.0)

Sperry Research was developing a process for extracting geothermal energy which took advantage of gravity, the Gravity Head Energy Conversion System. Advantageous use of gravity promised to yield increased energy extraction efficiencies. The objective of this miniproject was to identify elastomers that were compatible with the system fluids brine, Freon 114®, and synthetic oil.

Immersion tests were already run under the cognizance of Sperry. L'Garde's existing experience base was added to the Sperry information and a set of candidate elastomer compounds were selected for further evaluation in simulated seal tests.

In several instances, L'Garde's evaluation of elastomers as seals showed that immersion testing can be highly misleading for hostile geothermal environments. Immersion tests submit the elastomers to temperature and chemistry. An omission which is highly critical at high temperatures and/or pressures is the mechanical environment. Seals can be highly stressed through the setting process and/or by the differential pressure. Under hostile conditions, the mechanical environment becomes just as important as the thermochemical environment.

To provide the total environment including mechanical, L'Garde simulates actual field conditions in the laboratory. The Sperry candidates were tested as static O-ring seals in an existing test rig. Tests were run in Freon 114® and Mobil SHC 224/234 oil at 182C (360F) and 238C (460F) at 10.3 MPa (1500 psi) differential pressure for 140 hours. Because of the limited budget and significant prior data in brine, brine was not included in this testing.

In oil the two best elastomers were both EPDMs, E962-85 and Y267. E962-85 made by Parker Seal is based on Y267 EPDM. This conclusion was not expected in that EPDMs swell and lose strength and hardness in hydrocarbons, and the Y267 technology is not excepted though it is less vulnerable. This was one of the first instances where unexpected conclusions were revealed by proceeding beyond immersion tests into a more complete simulation of operational conditions.

The tests in the Freon 114® were inconclusive. It had a severe effect on all eight compounds; only two fluorelastomers, 3M Fluorel® Compound C and Parker V747-75, survived the 140-hour test. The two survivors were badly cracked. More work is required to provide the confidence required to identify an elastomer for Freon 114®

1.2.3 Electrical Elastomer Compounds (Section 4.0)

High temperature has been the nemesis for geothermal logging. In many instances logging was simply avoided. However, because of the research nature of the Los Alamos National Laboratory (LANL) Hot Dry Rock (HDR) Project, obtaining data at temperatures to 320C (608F) was imperative so the challenge was met head-on. A critical factor in solving the high temperature logging challenge was to provide seals that could withstand the environment. LANL sponsored L'Garde to evaluate the incorporation of the

Y267 EPDM technology into their tools.³ This effort turned out to be quite successful and as a consequence, LANL converted all their logging tool O-ring seals to Y267 EPDM.

Having solved the most critical problem, O-ring seals, one of the next most critical problems was the elastomeric boot that protects feed-through connectors. This problem could not be solved with Y267 EPDM because the boot needed to be highly electrically resistive and Y267 EPDM is not.

A miniproject was set up to modify the Y267 EPDM to obtain the proper electrical characteristics. Compound modifications were formulated, and resistivity was evaluated at room temperature and 50% relative humidity as well as at elevated temperatures to 288C (550F). Based on this limited effort two compounds (323 and 331) were selected for application testing at Los Alamos. 323 used an Austin Black® (bituminous coal fines) filler and 331 used FEF carbon black and #1 Barytes (natural barium sulfate). They provided resistivities of 3×10^9 to 7×10^{14} ohm-cm and 2×10^{11} to 6×10^{11} ohm-cm respectively over the range of conditions tested, however, 331 was not tested under all conditions.

This task was completed with the fabrication of connector boots which were shipped to LANL for testing. The results of that testing were not available at the conclusion of this overall project. Because this effort was limited, the delivered compounds were not fully optimized.

1.2.4 Explosive Stimulation Experiment Seals (Section 5.0)

Union Oil, Physics International, and Rocket Research engaged in a project with the DOE through Los Alamos National Laboratory (LANL) to explosively stimulate a geothermal well. The objective of the project was to demonstrate that such a stimulation could be accomplished safely. The Hitex II® explosive, the detonator, and the overall containment were developed for this experiment.

The detonator and containment vessels both used elastomeric O-rings for closure seals. Tests in the LANL autoclave at 260-275C (500-525F) indicated problems with commercially available Teflon® and Viton® seals. At this point a few weeks prior to the anticipated experiment date, L'Garde was contacted and the newly developed elastomers were considered.

255 Viton, 266 EPDM/Viton® blend, Y267 EPDM, and the commercial Viton that was originally planned for use were evaluated in the Hitex II®

explosive. A 24 hour exposure at 246C (475F) was run and degradation in physical properties evaluated. Of the candidates tested, the Y267 EPDM clearly sustained the Hitex II® exposure better than the other candidates. Furthermore, it was known that it is superior in a steam environment.

Molds for the required O-rings and the Y267 EPDM O-rings were fabricated and provided for the explosive experiment. Two downhole tests were run, a subscale preliminary test to check out the system, and the actual full-scale experiment. Both devices detonated on schedule, 48 hours after timer initiation, providing the basis for the conclusion that the O-rings sealed successfully.

1.2.5 Brief Minisupport Tasks (Section 6.0)

Several minor support events were made possible through the GEM-TA Program. These events are summarized in Section 6.0.

A. Operator Briefings (Section 6.1)

A major emphasis on the GEM-TA Program and especially on these minisupport tasks was to gain maximum impact for each dollar spent. It was determined that the geothermal community was not taking advantage of the GEM elastomer technology anywhere near its potential extent. This meant that operators were unnecessarily experiencing and dealing with problems and failures that could be eliminated by using the Y267 EPDM technology.

A chain of several organizations are involved from the point of manufacture of an elastomeric component to its end use. This chain may include some or all of the following:

- Elastomer compounder
- Elastomeric part manufacturer
- Subcomponent manufacturer
- Tool or component manufacturer
- Service Company
- Operator

Poor or good performance of any tool or component is ultimately reflected in the cost to the end benefitee, the operator. If a failure occurs he ultimately pays if not directly in dollars, by not accomplishing the objective, by lost production time, by extended rig time, etc. In the same manner, good performance will ultimately result in savings to the operator.

In that the operator is the clear benefitee, it was judged most efficient to make him aware of the GEM elastomer technology. In the ideal scenario the operator becomes convinced of the advanced performance provided by the GEM elastomers, and when he contracts for services or buys equipment he contractually obligates his suppliers to incorporate Y267 EPDM for example. The operator is the only one in a position to force the entire chain to respond in a ripple-down or domino fashion. Hence, the strategy of informing the operators was hoped to result in maximum increased use of the GEM elastomer technology for the small budget available.

Eighteen organizations in California, Utah, and Texas were briefed on the status of the GEM elastomer technology and several case history experiences.

B. Cementing Wiper Plug (Section 6.2)

Los Alamos National Laboratory (LANL) experienced more than their share of cementing problems on the Hot Dry Rock (HDR) Project because of the extreme temperatures up to 320C (608F). One of the problems associated with the cementing is the elastomeric wiper plug. It provides a moving barrier or separation plug which follows the cement down into the well and prevents contamination by the follow-up fluid column.

LANL tested several available wiper plugs in their autoclave at 280C (536F) and all suffered degradation with one disintegrating so completely it disappeared. After months of requesting Y267 EPDM wiper plugs from industry, Dowell committed to provide them to the HDR Project. Dowell obtained uncured Y267 EPDM from L'Garde, and had L'Garde post cure the molded wipers.

The Y267 EPDM wipers showed no degradation in the autoclave tests. A successful cement job resulted at 275C (525F), however, the plug was not located during drill out so its performance could not be confirmed.

C. Spontaneous Technology Transfer (Section 6.3)

The GEM-TA Program provided the means to answer various spontaneous inquiries regarding the GEM elastomers technology. Numerous telephone, letter, and first-person inquiries were satisfied by providing various information. 26 different types of organizations were provided information. Most were American organizations, however, inquiries also came from six foreign countries.

D. Presentation of Papers at Technical Symposia (Section 6.4)

During the course of the GEM-TA Program, papers describing GEM elastomers status and case histories were presented at three technical symposia. The symposia are as follows:

- GRC Annual Meeting, October 1981, Houston.
- Sandia High-Temperature Electronics and Instrumentation Conference, December 1981, Houston.
- IADC/SPE Drilling Conference, February 1983, New Orleans.

2.0 LINESHAFT PUMP BEARINGS

Section 2.0 summarizes a small effort performed to investigate the potential for elastomeric lineshaft bearings for downhole geothermal pumps.

2.1 PUMP BEARING BACKGROUND

Geothermal reservoirs with insufficient pressure to sufficiently deliver the energy to the wellhead need a pump that will push the fluid from the well. Surface pumps which pull or suck the fluid from the well are not practicable as cavitation or flashing of the fluid will occur, hence, the pump must be located sufficiently deep in the well. Two basic centrifugal pump configurations are candidates for this application, one where the motor is immersed along with the pump and the other where the motor is mounted at the wellhead with a driveshaft or lineshaft that extends down to the pump.

Both types of pumps have been experiencing problems in geothermal applications and some kind of reliable pump is needed for applications such as the binary power plants in Heber and El Centro. The submersible electric pumps have problems with fluid getting into the motor or cables resulting in electrical shorts. The lineshaft pumps have problems with lineshaft bearings working at geothermal temperatures. In about 1980, the DOE identified the pump situation as a critical problem that needed a solution. Some development effort has been expended on submersible electric and lineshaft pumps.

A minor effort was spent specifically on applying GEM Y267 EPDM⁴ technology to the geothermal lineshaft pump bearing problem. The DOE funded L'Garde to research the subject, and based on those findings, modify the Y267 EPDM formulation and mold bearings for test at Solar Turbines in San Diego. This effort was intended to be a minimal high-risk, bare-bones effort which hopefully would give some indication as to the feasibility of the GEM Y267 EPDM technology raising the rubber bearing capability from its everyday common usage level of 50C (122F) to geothermal temperatures on the order of 150-200C (302-392F).

2.2 RUBBER LINESHAFT BEARINGS

Rubber or elastomeric bearings have been used successfully for

decades. The application was discovered by a gold miner at about the turn of the century when an emergency required that he jury rig a pump with a failed bearing. In desperation he longitudinally slit a rubber hose and replaced the metal journal bearing with the hose. Not only did the hose bearing work, but it significantly surpassed the performance of the metal bearing it replaced.

Experience over the years indicates that there is probably no better bearing than an elastomeric one that performs with low viscosity lubricants and grit. The reason that rubber bearings can tolerate grit is due to its compliant quality. If a particle gets between the shaft and the bearing, the rubber allows the particle to be imbedded into it and to be rolled across its surface to a longitudinal flushing/lubricating flute where it is flushed out of the bearing. The rolling action of the particle across the rubber occurs without damage or cutting, hence, the copyrighted trade name for Goodrich bearings is "Cutless"® bearings. The predominant applications are ship propeller shafts and water pump lineshafts. Water pump bearings are reported to commonly perform maintenance free in excess of ten years with cases exceeding 20 years. The ship propeller shaft application has evolved several advantages over the years including the following:

- Effective for use with high speed shafts because they allow the shaft to turn on its center of gyration even when it does not correspond to its geometric center. This has also turned out to be important when the center of gyration continuously changes, as will happen if one propeller blade is damaged or broken off.
- The bearing friction is insensitive to large changes in clearance on the order of a factor of 10 or more.
- Successful operation is possible even with bent shafts.

The geothermal feed pump application is the same as the water pump except for two important differences -- temperature and precipitated mineral particulates in addition to sand. The temperature creates the greatest challenge since most rubber will not hold up under geothermal conditions. But even with the problems that are being experienced with geothermal lineshaft bearings;

- the years of success enjoyed by rubber bearings in propeller shaft and water pump lineshaft applications,

- the forgiving nature of rubber bearings,
- the low viscosity particulate containing nature of the produced fluid, and
- the success experienced with the DOE/L'Garde Y267 EPDM in other geothermal, and high-temperature oil and gas seal applications;

it follows that rubber or elastomeric bearings for geothermal lineshaft pumps deserve significant evaluation of feasibility and development. Additional effort beyond the scope of the Solar/L'Garde mini-evaluation reported here is warranted.

2.3 TEST SPECIMENS

Based on the research, L'Garde fabricated a mold sized to provide the desired clearances at the operating temperature of 182C (360F). In addition, compound 317 EPDM was formulated to provide a 70-75 Shore A hardness at room temperature, the desired hardness for the bearing. Both bearings and standard 152.4 x 152.4 x 1.905 mm (6 x 6 x .075 inches) ASTM test slabs made from 317 and Y267 EPDMs were provided to Solar Turbines for evaluation and testing. The 317 EPDM compound was the "first try" bearing compound and the Y267 EPDM was the packer seal compound developed in 1979 which served a control function. The bearings were sized for the Solar lineshaft bearing simulator test rig and the ASTM slabs were provided for autoclave immersion testing, both in brine.

Three Y267 and five 317 EPDM bearings were delivered along with two test slabs on each compound. The nominal room temperature configuration of the bearing is shown in Figure 2-1.

Figure 2-1. Test Bearing Configuration, Dimensions in mm and (inches)

This bearing is sized for a nominal 25.4 mm (1.000 inch) shaft. Solar cut the bearings to a length of about 69.85 mm (2.75 inches) to fit the bearing housing finally used.

The rubber parts were delivered with the following precautions:

- Mating shaft surface should be "ground and polished" or "piston finished".
- Do not allow oil or hydrocarbon substances to contact the rubber part -- it will swell.
- Center bearing in holder lengthwise so it can expand equally in both directions.
- Do not apply bearing load over 241 nkN/m^2 (35 npsi).
- If lubricant is needed for installation, recommend water or saliva.

2.4 TEST CONDITIONS

Solar Turbines ended up running two 317 and two Y267 EPDM bearings in brine. The first set, one of each compound, was run at 93C (200F) and the second at 182C (360F). They were run simultaneously in two independent twin test rigs at 1700-1800 RPM and a constant 445N (100 lb.) side force. The 93C tests were continuously run for 100 hours and the 182C tests for 50 hours. Shorter tests were run at 182C for economic reasons because the leakage at the higher pressure required round-the-clock manning.

Portions of one 317 and one Y267 EPDM test slabs were immersion aged for 1000 hours at 182C (360F) in brine. These were provided back to L'Garde for tensile measurements.

2.5 TEST RESULTS

Throughout all the dynamic testing of the rubber bearings there was no indication of any anomalies while the tests were running. The operation was smooth and quiet.

On disassembly of the bearings in the 100 hr. 93C test, no damage

to the bearing had occurred to either the 317 or Y267 EPDM bearings. This in itself is a worthy accomplishment in view of the elevated temperature and 445N (100 lb.) side force. Figure 2-2 shows the two post mortem bearings placed side-by-side.

Even though they were apparently running nominally, both bearings which were run at 182C (360F) suffered some mechanical damage during the 50 hr. test. The Y267 EPDM bearing was more severely damaged. It appears as if rubber was chunked out to a depth of 1.829 mm (0.072) inches below the bearing flat. Enough friction was created between the bearing and the shaft to cause the seal to rotate in its holder. This is evident from the circumferential cut from the set screw. Figure 2-3 shows two halves of the bearing, one an ID view and one an OD view.

The 317 EPDM bearing shows less damage than the Y267. The evidence indicates that material was removed through well controlled abrasion, i.e. the line of demarcation where the material is removed in a smooth elliptical shape as opposed to the discontinuous pattern on the Y267 EPDM which indicates chunking, to a depth of 0.965 mm (0.038 inches). The friction between the shaft and the bearing was never high enough to cause rotation of the bearing in the holder. Figure 2-4 shows the two halves of the 317 EPDM bearing, one an ID view and one an OD view.

Table 2-I summarizes the dynamic test results.

TABLE 2-I. SUMMARY OF DYNAMIC TESTS AT SOLAR TURBINES

Compound Part No.	Temp. C (F)	Duration Hours	Constants	Comments
Y267	93 (200)	100	Brine	No apparent damage or degradation of bearing.
317	93 (200)	100	~1700 RPM	No apparent damage or degradation of bearing.
Y267	182 (360)	50	1" d. Shaft	Abrasion and chunking to depth of 0.072 inches.
317	182 (360)	50	100 lb. Force	Well controlled abrasion to depth of 0.038 inches.

Static immersion tests were run by Solar Turbines in autoclaves containing brine on both the Y267 and 317 compounds. L'Garde measured ultimate tensile properties and hardness of the virgin and chemically aged materials. Table 2-II summarizes those results. Both compounds show good hydrothermochemical stability after 1000 hours in brine. However, the data are inconsistent with earlier measurements indicating the retained properties after ageing may be even better than those shown. Table 2-III

Figure 2-2. Y267-CXIX-2 and 317-II-2 Bearings In As-New Condition After Testing At 93C (200F) For 100 Hours.

Figure 2-3. Y267-CXIX-3 Bearing Showing Chunking After Testing At 182C (360F) For 50 Hours.

Figure 2-4. 317-II-4 Bearing Showing Well Controlled Abrasion After Testing At 182C (360F) For 50 Hours.

TABLE 2-II. SUMMARY OF STATIC IMMERSION TESTS AT SOLAR TURBINES

Compound/Part No.	Constants	σ ult., psi			ϵ ult., %			Shore A		
		V	CA	CA/V,%	V	CA	CA/V,%	V	CA	CA/V,%
Y267-CXIX-2	182C (360F)	1935	1377	71	220	153	70	91.5	89	97
	1000 hours									
317-II-2	Brine	1273	1345	106	286	271	95	74	75	101

V = Virgin

CA = Chem Aged

TABLE 2-III. COMPARISON OF STATIC IMMERSION TESTS ON ECT PROGRAM

Compound/Duration	Constants	σ ult., psi			ϵ ult. psi			Shore A		
		V	CA	CA/V,%	V	CA	CA/V,%	V	CA	CA/V,%
Y267										
0 months	Syn. Brine	1945			240			92		
2 months	191C (375F)		1725	89		200	83		91	99
6 months			1695	87		100	79		92	100

V = Virgin

CA = Chem Aged

are some comparable measurements made on the BNL/L'Garde ECT Program. Here the specimens were immersed in synthetic geothermal fluid of the following constituency:

H₂S 300 ppmCO₂ 1000

NaCl 25,000

H₂O balance

Comparing the same Y267 EPDM compound, the ECT tests show better retention of properties for two months and even six months at a slightly higher temperature as compared to the 1000 hours or 1.5 months in Table 2-II. In addition, the slightly higher temperature (8C (15F)), according to

Arrhenius, gives an equivalent of nearly four months and twelve months at the lower temperature. There are a number of potential reasons for this apparent inconsistency, such as:

- Difference between Solar brine and L'Garde synthetic geothermal fluid.
- Defective constituents or processing of Y267 EPDM.
- Autoclave temperatures or conditions different from that indicated.

In any event, the aged properties of both Y267 and 317 EPDM both tested excellent and may be even better.

2.6 CONCLUSIONS REGARDING ELASTOMERIC LINESHAFT BEARINGS

Given the fact that a significant segment of the geothermal resource requires a reliable downhole pump for energy production and there is no clear solution to this need, developing solutions is warranted and the lineshaft pump is an extremely pertinent contender. The results of the limited investigation of the feasibility of applying elastomeric bearings to the geothermal lineshaft pump application are positive, or at the very worst are not negative, and indicate further investigation is warranted. This is especially evident when considering the following extremely pertinent facts:

- No development occurred -- there was some limited research and one "best-guess" compound was formulated for the lineshaft bearing tests.
- The 100 hour test with a constant 445 N (100 lb.) side load at 93C (200F) was completely successful, "best-guess" compound bearing came out as new.
- The 50 hour test (reduced because 24 hour manning required at higher pressure because of leakage) with a constant 445N (100 lb.) side load at 182C (360F) ran apparently trouble-free but on disassembly limited abrasion of the "best-guess" compound was observed.

- At 182C (360F) there was a definite improvement of the "best-guess" compound compared to the control the Y267 EPDM packer seal compound.
- The constant 445N (100 lb.) side load is more severe than actual operation where the side force would not build as high because of the compliant nature of rubber and would approach zero as the bearing wear relieved the shaft misalignment.
- Rubber bearings are in very successful common usage for lineshaft irrigation pumps, 10 to 20-year life is not uncommon (SAME EXACT CONFIGURATION AS GEOTHERMAL FEED PUMPS), and ship propeller shaft bearings.
- Rubber will tolerate grit such as sand in water, sea water, and geothermal fluid; where other materials won't.
- Rubber has good inherent damping qualities which will help keep undesirable shaft dynamics under control.
- Rubber bearings are extremely forgiving as witnessed over several decades in the ship propeller shaft application where conditions such as propellers with one blade broken off and bent shafts are tolerated.
- Rubber bearings provide the potential for a simple (economic) open lineshaft system with the bearings lubricated by the produced fluid. Magma's pumps have enclosed lineshafts and bronze bearings with 2 gallons per day of lubricating oil being pumped down from the wellhead.

3.0 ELASTOMER COMPATIBILITY FOR SPERRY GRAVITY-HEAD GEOTHERMAL ENERGY CONVERSION SYSTEM

Sperry Research was implementing a demonstration of the Gravity Head Energy Conversion System. They were having and anticipating elastomers problems with three basic seal types that they employed in their system: O-ring seals, sliding cup packer seals, and sliding lip type expansion type seals. Their maximum temperature is 182C (360F).

Comprehensive elastomer compatibility tests were needed for this application. The elastomeric components were critical to the operation of the system and their failure would imply shut down and work over of the turbine pump and heat exchanger that are located downhole. The DOE contracted L'Garde to perform the elastomer compatibility tests to supplement those previously performed by Sperry.

3.1 SUMMARY

In conjunction with Sperry Research Center, the test compounds, fluids and conditions were selected. These are listed in Table 3-I and 3-II. Each of the eight compounds were tested in two fluids and at two

TABLE 3-I. ELASTOMER TEST COMPOUNDS

Compound	Elastomer	Trade Name	Source
ON803	ethylene/acrylic	Vamac®	DuPont
Unknown	EP	Epcar®	Goodrich
Y267	EPDM	Nordel®	L'Garde
501	FKM	Viton®	L'Garde
C	FKM	Flourel®	3M
V747-75	FKM	Unknown	Parker
E692-85	EPDM	Nordel®	Parker
Unknown	EPDM	Unknown	Regal

TABLE 3-II. TEST CONDITIONS

Fluids:	Mobil SHC224/234 Oil Freon 114
Temperatures:	182C (360F) 238C (460F)
Pressures (O-ring):	Front - 10.3 MPa (1500 psi) Back - 0-.2 MPa (0-30 psi) vapor pressure
Time:	140 hours

temperatures. Tests were not done in brine due to economic limitations and substantial data already existed. It was known that the Y267 type EPDM is clearly superior for brine. The test equipment and methods were those developed under the ECT Program.⁵

All eight compounds performed well in the Mobil SHC224/234 oil. There was some swelling of the EPDMs but it did not hinder their performance as static seals. There was no cracking, nibbling, extrusion or failure of any of the eight.

Based upon the degree of permanent set, the compounds after withstanding Mobil SHC224/234 at 182C (360F) and 238C (460F) with a 10.3 MPa (1500 psi) differential pressure for 140 hours were ranked as follows:

<u>Rank</u>	<u>Compound</u>
1	Parker EPDM E962-85 (based upon L'Garde Y267 EPDM)
2	L'Garde Y267 EPDM
3	Parker Fluoroelastomer V747-75
4	Goodrich EP EPCAR®
5	L'Garde 501 Viton®
5	3M Fluorel Cpd C®
5	Regal EPDM
5	DuPont Vamac ON803®

Photos of the best and one of four "worst" are shown in Figures 3-1 and 3-2. The difference can be seen only in the shape of the cross section.

None of the eight compounds faired too well in Freon 114®. While all survived 140 hours at 182C (360F), only two survived 140 hours at 238C

Figure 3-1. Parker EPDM E962-85 Exposed to Mobil SHC224/234 Oil at 10.3 MPa ΔP for 140 Hours.

Figure 3-2. 3M Fluorel Cpd C[®] Exposed to Mobil SHC224/234 Oil at 10.3 MPa ΔP For 140 Hours.

(460F). According to the Arrhenius approximation, the latter condition is equivalent to 182C for nearly a year. Even the two survivors, Parker Fluoroelastomer V747-75 and 3M Fluorel Cpd C[®], were badly cracked.

The relative ranking follows, based upon degree of permanent set, time to failure and brittleness for the eight compounds after exposure to 182C (360F) and 238C (460F) Freon 114[®] for 140 hours with a 10.3 MPa differential pressure.

<u>Rank</u>	<u>Compound</u>
1	3M Fluorel Cpd C [®]
1	Parker Fluoroelastomer V747-75
3	L'Garde 501 Viton [®]
4	Parker EPDM E962-85
5	L'Garde Y267 EPDM
6	Regal EPDM
7	DuPont Vamac ON803 [®]
8	Goodrich EP EPCAR [®]

Of these, only the top three may work in Freon 114[®]. Use, however, for a year appears unlikely. Figures 3-3 and 3-4 are photos of one of the best and of the worst.

Under the prior ECT Program,⁵ Parker Fluoroelastomer V747-75 became brittle after five days in 191C (375F) brine, while L'Garde Viton 501[®] performed well in both 191C immersion and 191C-266C (375-510F) O-ring tests. 3M Fluorel Cpd C[®] was not tested in the ECT Program. Therefore, with the inclusion of the available brine data, there is insufficient data to identify an elastomer for a confirmation of Freon[®] and brine. In addition, the severe reaction of all candidates to Freon 114[®], begs further testing before reliable candidates can be more confidently identified.

The following sections describe the test program in greater detail.

3.2 ELASTOMER COMPOUND SELECTION

Sperry Research Center and L'Garde personnel bilaterally selected the elastomer candidates, test fluids and test conditions. The fluids of interest were brine, Freon 114[®] and a Mobil Oil blend of 1 part SHC234 and 2 parts SHC224. There were insufficient funds for testing in three fluids

Figure 3-3. Parker Fluoroelastomer V747-75 Exposed to Freon 114[®] at 10.3 MPa ΔP For 140 Hours.

Figure 3-4. Goodrich EP EPCAR[®] Exposed to Freon 114[®] at 10.3 MPa ΔP For 140 Hours.

so brine was omitted. Considerable brine data were available from the ECT Program,⁵ therefore it was decided that such tests, though important, were the least necessary. Mustang Engineering supplied 5 gallons (0.019m³) of the oil blend, and the Freon was purchased.

The maximum operating pressure expected was 7.6 MPa (1100 psi.) It was decided to perform the simulation tests at 10.3 MPa (1500 psi) to provide some margin. The expected nominal temperature was 116C (240F) with excursions to no more than 182C (360F). The latter was selected as the minimum test temperature.

On the ECT Program, three 46-hour tests were run at different temperatures; tests at the two higher temperatures could be considered accelerated ageing tests. Sperry preferred longer test times and fewer temperatures. It was decided to run at 182C (360F) and 238C (460F); each test would take 140 hours. Per Arrhenius, 238C for 140 hours is equivalent to 182C for nearly a year.

The best elastomers indicated by the prior Sperry Research immersion tests were:

- DuPont Vamac 91680[®]
- DuPont Vamac ON803[®]
- Goodrich EP EPCAR[®]
- L'Garde Fluoroelastomer 501 Viton[®]
- 3M Fluoroelastomer Fluorel Cpd C[®]
- Parker EPDM E692-75
- Parker Fluoroelastomer V747-75
- Regal EPDM
- Regal Fluoroelastomer

To this list, L'Garde Y267 EPDM and 291 AFLAS[®] were added as potentials.

After reviewing the data, the following eight compounds were selected by L'Garde and Sperry.

- DuPont Vamac ON803[®]
- Goodrich EP EPCAR[®]
- L'Garde Y267 EPDM
- L'Garde Fluoroelastomer 501 Viton[®]
- 3M fluoroelastomer Fluorel Cpd C[®]
- Parker Fluoroelastomer V747-75
- Regal EPDM
- Regal Fluoroelastomer

It was subsequently learned that Regal Fluoroelastomer and 3M Fluorel® Compound C were identical. It was therefore decided to substitute Parker's compound based on Y267, E962-85, for the Regal Fluorel 114®.

L'Garde contacted the companies and they (except for Parker) shipped uncured rubber and curing instructions to L'Garde. L'Garde molded the O-rings. Parker V747-75 O-rings were purchased from a distributor, and Parker furnished O-rings made from EPDM E962-85.

L'Garde also molded ASTM test slabs from the furnished uncured rubber, and conducted physical property tests on these and test slabs furnished by the suppliers. This provided basic data plus a means of assuring that our molding procedure produced results similar to the supplier's. Table 3-III contains the data.

TABLE 3-III. ELASTOMER PROPERTIES

Compound	Molded By	Specific Gravity	Tensile Strength MPa (psi)	Elong. @ Break %	Hardness Shore A
DuPont Vamac® ON803	L'Garde	1.23	16.3 (2360)	780	71
Goodrich EP EPCAR®	Goodrich	1.05	≥7.7 (≥1100)	>610	65
Goodrich EP EPCAR®	L'Garde	1.06	≥7.7 (≥1100)	>610	67
L'Garde EPDM Y267	L'Garde	1.12	11.9 (1720)	200	92
L'Garde Viton® 501	L'Garde	1.84	17.2 (2500)	210	84
3M Fluorel® Cpd C	3M	1.93	14.9 (2160)	190	94
3M Fluorel® Cpd C	L'Garde	1.94	13.4 (1940)	190	94
Parker Fluoroelastomer V747-75	Parker	1.88	15.6 (2260)	460	77
Parker EPDM E962-85	None	Should be similar to L'Garde EPDM Y267			
Regal EPDM	L'Garde	1.14	9.2 (>1330)	>610	75

3.3 TEST SETUP

Figure 3-5 shows the piping and instrumentation diagram of the test setup. The N₂ Tank 2 is the source for the front face pressure. Tank 2 drives the positive displacement pump which provides a 11.7 pressure amplification. This intensified pressure is transmitted by hydraulic fluid to the four accumulators. Each contains 115 cc of the test fluid, and serves one of the elastomers being tested.

Figure 3-5. O-Ring Test Piping and Instrumentation Diagram.

Should one of the compounds fail, its associated piston accumulator advances until it bottoms out, forcing its contents past the failed O-ring into the back pressure accumulator. The back face fluid is held at a constant pressure by the N₂ Tank 1.

The test section (Figure 3-6) is heated by eight band heaters on the outside of the Inconel 600 pressure vessel. Temperature feed-back for control is provided by a single resistance thermometer. The controller adjusts the supply voltage to the heaters providing continuous heating.

Figure 3-6 shows eight O-rings -- two for each of four individual test sections. The high pressure test fluid comes up four individual passages in the inner stainless steel cylinder which lead to their respective smaller passages in the stainless steel O-ring holder between two O-rings of the same compound. At failure, the fluid leaks to the center of the O-ring holder and out through the upper closure.

The as-measured dimensions of the O-ring interfaces are provided in Figure 3-7. The downstream or the clearance gap the rubber must bridge is 0.08 mm (0.006 in) on the diameter.

Figure 3.7. As-Measured 2-329 O-Ring Groove Dimensions In Inches

Figure 3-6. Test Section.

Instrumentation consists of eight pressure gages (Figure 3-5), six thermocouples (Figure 3-6), and four accumulator piston position sensors (Figure 3-5). The radial distances from the thermocouples to the center of the O-ring grooves are 41.9 and 6.9 mm (1.65 and 0.272 in) for the outer and inner thermocouples, respectively. The thermocouples and pressure gages are read and recorded manually. The piston positions are recorded on a strip chart so that leak rate and/or time of failure is recorded.

Photographs of the set-up are shown in Figures 3-8 through 3-12.

3.4 TEST PROCEDURE

The following is the procedure used for the O-ring tests.

- a. Install the O-rings on the holder and install the holder in the test section.
- b. Evacuate the system, and back-fill with the test fluid -- such that the fluid is on both sides of the O-rings.
- c. Turn on heaters.
- d. Apply the front face pressure after about 1-1/2 hours of heating, approximate time to reach the test temperature.
- e. Monitor the test and make adjustments.
- f. Depressurize for 5 minutes three times during the 140 hours.
- g. Turn off heaters 140 hours after Step d, and air cool with fan; allow pressure to decay due to reduced temperatures.
- h. Repressurize when the O-ring is approximately 90C (200F) (approximately 1-1/2 hours after start of cooling).
- i. Depressurize after 1/2 hour or more.
- j. Remove O-ring holder, record observations, and photograph.

Figure 3-8. O-Ring Test Facility.

Figure 3-9. Test Section Area, Pressure Amplifier and Back Pressure Accumulator.

Figure 3-10. Test Section.

Figure 3-11. Temperature Controls and Strip Chart Recorder.

Figure 3-12. Front Face Pressure Accumulators.

3.5 O-RING TEMPERATURES

While the nominal test temperatures are 182C (360F) and 238C (460F), actual temperatures are different because of a) temperature changes with time, and b) temperature gradients along the test section. Six thermocouples were used to determine actual average temperatures. Four were located under the band heaters and two are located close to the O-rings as previously illustrated in Figure 3-6.

T6, which is 6.9 mm (0.272 in) outboard of the O-rings and near the center of the 8 O-rings, was used to plot the temperature time history shown in Figures 3-13 and 3-14. Readings were taken manually. All readings are connected by straight lines.

It is seen in Figure 3-13 that the EPDM/EP 238C test was terminated at 144 hours instead of 140 hours. Figure 3-14 shows that one of the 238C tests did not get up to the proper temperature for a day. This occurred because the digital thermometer was set for the wrong type of thermocouple, and showed readings approximately 55C higher than actual.

Average T6 temperatures were calculated from Figures 3-13 and 3-14 and are shown in Table 3-IV.

TABLE 3-IV. TIME AVERAGED TEMPERATURES

	Freon 114®		Mobil SHC224/SHC234	
Desired Temperature	FKMs Vamac®	EPDMs EP	FKMs Vamac®	EPDMs EP
182C (360F)	184C	185C	183C	185C
238C (460F)	231C	241C	239C	240C

The geometric temperature gradients are reconstructed in Figures 3-15 and 3-16. T1 through T4 are 41.9 mm (1.65 in) from the O-ring groove cylinder, while T5 and 6 are 6.9 mm (0.272 in) away. The latter are close enough to the O-rings to represent their temperatures. The readings plotted are those taken near the end of the 140-hour test.

It was assumed that the temperature gradient between T5 and T6 was

Figure 3-13. Mobil Oil Test T6 Temperature History

Figure 3-14. Freon 114[®] Test T6 Temperature History.

Figure 3-15. Mobil Oil Test Temperature Gradients.

Figure 3-16. Freon 114® Test Temperature Gradients.

linear. Extrapolation from T6 was done parallel to the T1-T3 temperatures.

The temperature changes for Figures 3-15 through 3-16 for each compound were adjusted for the average temperatures formerly discussed to produce the values in Table 3-V. The compounds are listed in the order of

TABLE 3-V. O-RING RECONSTRUCTED AVERAGE TEMPERATURES

Test Fluid	Freon® 114		Mobil SHC224/234	
Desired Temperature, C (F)	182 (360)	238 (460)	182 (360)	238 (460)
Actual Temperature, C (F)				
E962-85	185 (365)	241 (466)	185 (365)	240 (464)
501	185 (365)	231 (448)	183 (361)	239 (462)
Regal EPDM	184 (363)	239 (462)	183 (361)	237 (459)
Y267	181 (358)	237 (459)	182 (360)	235 (455)
V747-75	183 (361)	228 (442)	180 (356)	237 (459)
Cpd C	183 (361)	228 (442)	181 (358)	234 (453)
EP	180 (356)	234 (453)	178 (352)	231 (448)
Vamac® ON803	179 (354)	222 (432)	175 (347)	231 (448)
Average Temperature, C (F)	183 (361)	233 (451)	181 (358)	236 (457)
Temperature Spread, C (F)	6 (10.8)	19 (34.2)	10 (18)	9 (16.2)

approximately decreasing temperature. The absolute temperature is not as important as temperature spread because the compounds are being compared to each other. The spread ranges from 6C to a rather large 19C. This was taken into account in the evaluation, but did not affect the rankings.

3.6 PRESSURE

The nominal front face pressure was 10.3 MPa (1500 psia), and the back face pressure was the vapor pressure of the fluid: 0.2 MPa (30 psia) for Freon 114 and 0.1 MPa (14.7 psia) for oil.

Due to the high design differential pressure, the displacement pump has a Chevron seal pack which has fairly high friction. It was observed during testing that the liquid pressure would slowly increase with time until the seal friction was overcome, and the displacement pump would back off. The nitrogen pressure would not change with time unless adjusted. It is unknown why the test fluid pressure would gradually increase.

Figures 3-17 and 3-18 provide the pressure histories as recorded. Data are connected by straight lines and it is unlikely that continuous curves would look like those in the figures. These curves provide the probable pressure extremes of 8.5 to 11.5 MPa. The test equipment pressure tolerance is therefore about $\pm 15\%$.

Marked on Figures 3-17 and 3-18 (as well as on Figures 3-13 and 3-14) are the times at which the system was depressurized for about five minutes. This was done to pressure cycle the O-rings and encourage failure.

3.7 TEST RESULTS AND EVALUATION

The evaluation was based upon leakage and/or catastrophic failure during the test, and physical condition of each O-ring after the test. Provided below under each subsection for a particular fluid is a table that summarizes the test observations. Given are the O-ring test temperatures as calculated in Section 3.5, and the O-ring post-test thickness to give an indication of swell or deformation (original thickness was 5.3 mm). The conditions described under "Permanent Set" are defined as follows:

None	-- Circular cross section
Slight	-- Indication of flattening
Moderate	-- Downstream side flat
Severe	-- Three flat sides

The comments splitting, cracking, extruding, nibbling, catastrophic failure, et cetera are self-explanatory. Catastrophic failure is considered the worst condition where the O-ring could no longer hold pressure. Nibbling is progressive breakage of surface particles of rubber from the low pressure side with the residue ending up downstream. Extruding indicates evidence that the elastomer was forced into the downstream gap and permanent deformation or damage resulted.

3.7.1 Mobil SHC224/234 Oil

Table 3-VI summarizes the results. There were no failures, or cracked or brittle specimens. The only damage was various degrees of permanent set. Also the EPDMs swelled more than other compounds.

Figure 3-17. Mobil Oil Test Pressure Histories.

Figure 3-18. Freon 114® Test Pressure Histories.

TABLE 3-VI. MOBIL SHC224/234 OIL TEST OBSERVATIONS

Compound	Temp C(F)	X Section Dia, mm (in)	Permanent Set ?	Comments
3M Fluorel® Cpd C	181 (358) 234 (453)	5.6 (.220) 5.6 (.220)	Moderate Severe	No other visible change No other visible change
L'Garde 501 Viton®	183 (361) 239 (462)	5.5 (.217) 5.5 (.217)	Moderate Severe	No other visible change No other visible change
Parker FKM V747-75	180 (356) 237 (459)	5.4 (.213) 5.5 (.217)	Slight Moderate	No other visible change No other visible change
DuPont Vamac® ON803	175 (347) 231 (448)	5.5 (.217) 5.6 (.220)	Moderate Severe	One ring rolled (during installation?) No other visible change
L'Garde Y267 EPDM	182 (360) 235 (455)	5.7 (.224) 5.8 (.228)	Slight Moderate	No other visible change No other visible change
Parker EPDM E962-85	185 (365) 240 (464)	5.6 (.220) 5.7 (.224)	None Slight	No other visible change No other visible change
Regal EPDM	183 (361) 237 (459)	6.1 (.240) 6.2 (.244)	Moderate Moderate	No other visible change No other visible change
Goodrich EP EPCAR®	178 (352) 231 (448)	6.0 (.236) 6.1 (.240)	Slight Moderate	No other visible change No other visible change

The compounds were ranked as follows based upon the degree of permanent set:

<u>Rank</u>	<u>Compound</u>
1	Parker EPDM E962-85 (based on L'Garde Y267)
2	L'Garde Y267 EPDM
2	Parker Fluoroelastomer V747-75
4	Goodrich EP EPCAR®
5	L'Garde 501 Viton®
5	3M Fluorel Cpd C®.
5	Regal EPDM
5	DuPont Vamac ON803®

EPDM is generally not recommended for use with oil because of the measurable degradation in physical properties observed after immersion tests. However, in spite of these physical property changes E962-85 and Y267 performed in a superior manner as a static seal.

This perhaps controversial conclusion was first made during the ECT Program.⁵ Long-term immersion tests (up to 6 months) showed that good EPDM compounds stopped degrading within a few days, while good fluoroelastomers,

which are generally the unquestioned recommendation for high temperature hydrocarbon applications, continue to degrade with time. These data indicate that Y267 EPDM static seals will outlast fluoroelastomer static seals in the oils tested at high temperatures.

Photos of all the test O-rings are provided in Figure 3-19.

3.7.2 Freon 114[®]

Table 3-VII summarizes the results. While no compound failed at

TABLE 3-VII. FREON 114[®] TEST OBSERVATIONS

Compound	Temp C(F)	X Section Dia, mm (in)	Permanent Set ?	Comment
3M Fluorel [®] Cpd C	183 (361) 228 (442)	5.7 (.224) 6.1	Slight Moderate	No other visible change. Extensive splitting.
L'Garde 501 Viton [®]	185 (365) 231 (448)	5.6 (.220) 6.0 (.236)	Slight Severe	No other visible change. Extensive cracking, slight extrusion. Failed during cool-down @ 141 hours.
Parker FKM V747-75	183 (361) 228 (442)	5.6 (.220) 6.1 (.240)	None Moderate	No visible change. Slight nibbling, extensive splitting.
DuPont Vamac [®] ON803	179 (354) 222 (432)	5.7 (.224) ---	Moderate Severe	Minimal cracking. Very brittle, 15 gm/day leak rate at 24 hours. Failed @ 78 hours.
L'Garde Y267 EPDM	181 (358) 237 (459)	5.8 (.228) ---	Moderate Severe	No other visible change. Brittle, shrunk. Failed during cool-down at 141 hours.
Parker EPDM E962-85	185 (365) 241 (466)	5.6 (.220) ---	None Severe	No visible change. Failed during cool-down at 142 hours.
Regal EPDM	184 (363) 239 (462)	6.1 (.240) ---	Moderate Severe	No other visible change. 25 gm/day leak rate at 59 hours. Catastrophic failure at 97 hours.
Goodrich EP EPCAR [®]	180 (356) 234 (453)	6.1 (.240) ---	Moderate Severe	Some cracking. Very soft and gummy, 5 gm/day leak rate. Catastrophic failure at 76 hours.

182C (360F), all but two failed at the higher temperature. This indicates that few or none will last a year at 182C and with a differential pressure of 10.3 MPa (1500 psi).

Parker EPDM E962-85

L'Garde Y267 EPDM

Parker Fluoroelastomer V747-75

Goodrich EP EPCAR®

Figure 3-19. O-Rings After Testing With Mobil SHC224/234 Oil.

L'Garde Viton 501®

Regal EPDM

DuPont Vamac ON903®

3M Fluorel Cpd C®

Figure 3-19 (concluded). O-Rings After Testing With Mobil SHC224/234 Oil.

The compounds were ranked as follows:

<u>Rank</u>	<u>Compound</u>
1	3M Fluorel Cpd C [®]
1	Parker Fluoroelastomer V747-75
3	L'Garde 501 Viton [®]
4	Parker EPDM E962-85
5	L'Garde Y267 EPDM
6	Regal EPDM
7	DuPont Vamac ON803 [®]
8	Goodrich EP EPCAR [®]

Parker Fluoroelastomer V747-75 and 3M Fluorel Cpd C[®] are ranked best because they did not fail. L'Garde 501 Viton[®] is ranked third because it did not become brittle or soften as the other lower ranking compounds did.

Parker EPDM E962-85 and L'Garde Y267 EPDM are ranked 4 and 5, respectively, because failure occurred only during cool-down after the 240C (460F) test. The Parker compound (which is based on Y267 technology) appears slightly better than L'Garde's because it showed no permanent set after the 182C (360F) test.

The 6th, 7th, and 8th ranks of Regal EPDM, DuPont Vamac ON803[®] and Goodrich EP EPCAR[®], respectively, are based primarily on time to leakage and time to catastrophic failure.

However, only the three fluoroelastomers should be considered for use with hot Freon 114[®] because the other five compounds became either brittle or softened. None are likely to last a year in actual service, however, and further testing and evaluation is justified to better define a practical approach.

Photos of all the compounds are shown in Figure 3-20.

3.8 RECOMMENDATIONS

The tests showed that there are good elastomers candidates for hot Mobil SHC224/234 oil, but none were identified for hot Freon 114[®] or a hot Freon 114[®]/brine environment.

Parker Fluoroelastomer V747-75

3M Fluorel Cpd C®

L'Garde 501®

Parker EPDM E962-85

Figure 3-20. O-Rings After Testing With Freon 114®

L'Garde Y267 EPDM

Regal EPDM

DuPont Vamac ON803[®] Goodrich EP EPCAR[®]Figure 20 (concluded). O-Rings After Testing With Freon 114[®]

It is recommended that an elastomer be developed for use in a combination of Freon 114® and brine. The tests would indicate that a fluoroelastomer polymer would be the best candidate for such work.

Since the expense of development may be prohibitive, additional testing may be cost effective:

- a. If a life of less than a year is satisfactory, one of the three fluoroelastomers might be acceptable if they can handle brine for an adequate time. It is recommended that similar O-ring tests be conducted in 182C and 238C brine of the three fluoroelastomers.
- b. Freon 114® appears to present an intractable environment to elastomers. Therefore, it is recommended that Sperry review their design to determine if other thermodynamic fluids may be considered. If so, it is recommended that this test program be repeated for those fluids.

4.0 ELECTRICAL COMPOUNDS FOR LOGGING TOOLS

This section describes the brief development of modified Y267 EPDM compounds for electrical applications.

4.1 BACKGROUND

The Hot Dry Rock (HDR) Project is being implemented for the Department of Energy by Los Alamos National Laboratory (LANL). As the name implies, HDR involves extracting geothermal energy from dry granitic formations without the benefit of a coincident natural water supply to provide the media with which to extract the heat.

As with other types of geothermal resources, the HDR project is faced with deploying logging tools and making measurements in very hot wells. However, with the HDR project there is need for gathering much more information because it is a research project with an objective to prove the feasibility of the concept. In addition, the temperatures are very high, 320C (608F), rendering the data acquisition intractable.

The need for making measurements in that hostile environment has forced LANL to expand the state-of-the-art to satisfy that requirement. Elastomeric seals were a primary weakness in their logging tools. They investigated the compatibility of the DOE/L'Garde elastomer development, Y267 EPDM.³ Y267 EPDM proved to provide a significant improvement in their hostile environment logging capability. LANL has since converted all their logging tool seals over to Y267 EPDM technology except for those at electrical interfaces. Logging at 260C (500F) is no problem now, where previously questionable reliability was experienced at 200C (400F).

Solving the primary seal problem, allows the tool to run longer and cross into the damage threshold for the next most vulnerable component. One of the next thresholds is associated with the elastomeric boots that slip over feed-through connectors.

Typically if an electrical conductor needs to feed through or pass through a pressure-tight bulkhead, it is done with a feed-through connector. A typical connector concept is similar to a sparkplug where the conductor is potted in a ceramic which is attached to a screwed metal interface with a seal. Once the feed-through is screwed into place, the

electrical conductor is attached to the feed-through and that connection is often protected with an elastomeric boot or sleeve, again similar to the sparkplug.

It would seem to follow if Y267 EPDM works well for the primary seals, it should also work well for connector boots. This is true except for the fact that connector boots must have high electrical resistivity. Y267 EPDM is at the conductive end of the spectrum for elastomers. Figure 4-1 shows

Figure 4-1. Scale of Specific Resistance of Various Materials, "Engineering Uses of Rubber," edited by A. T. McPherson and Alexander Klemin, p. 280, Reinhold Publishing Corporation. 1956.⁶

the spectrum from very good conductors to very good insulators. Y267 EPDM has a resistivity or specific resistance of about 10^5 ohm-cm. For the connector boot application, it is desirable to have a material with a resistivity of at least 10^9 ohm-cm.

The primary objective of this task was to modify Y267 EPDM to improve its resistivity and thus develop a geothermal elastomer compound for electrical applications.

4.2 DEVELOPMENT APPROACH

This overall Program represented by this entire report was comprised of several mini-projects each addressing a specific geothermal elastomer problem. The need for an electrically insulative elastomer with good hydrothermochemical resistance like Y267 EPDM is well established. Even though the scope of this program was limited, and even though this mini-project within the overall program was even more limited, it was judged that an effort to modify Y267 and increase its resistivity was worthwhile even though the success risk was high due to the limited nature of the effort.

Several technical considerations were necessary. It has been well established that the electrical resistivity of polymers can be extremely sensitive to temperature. The scope of that temperature sensitivity is discussed in Reference 6. Figure 4-2 is a schematic diagram from

Figure 4-2. Schematic of Major Types of Resistivity-Temperature Curves⁶

Reference 6 showing typical temperature responses. Note that the High-Positive Temperature Coefficient (H-PTC) shows extreme sensitivity in the geothermal range of interest. The U.S. Geological Survey at Denver has measured temperature variability of geothermal connector boot elastomers.⁷ Figure 4-3 shows the temperature characteristics of the DOE/L'Garde Y267

Figure 4-3. Current Through Y267 EPDM at Temperature⁸

EPDM as measured by Sandia National Laboratory.⁸ They estimate that the resistivity of Y267 EPDM is about 2×10^5 ohm-cm at about 240C (464F).

Because of extreme temperature sensitivity that can occur with polymers, it becomes imperative to characterize any candidates for electrical applications at elevated temperatures. L'Garde set-up a test to measure DC volume resistivity of elastomers according to ASTM D257-78. The specific test procedure developed is included in this report as Appendix A.

This test measures the resistivity at 23C (73.4F) and 50% relative humidity. The test has a guarded electrode to prevent surface leakage currents from occurring. This is critical for sheet type specimens. The same apparatus is placed into a nitrogen purged oven instead of the humidity chamber to obtain resistivity measurements at elevated temperature.

The elastomer constituent which provides electrical conductance is the carbon black. Nonblack compounds using nonconductive mineral fillers such as clays are a common approach for electrical applications. In addition, proper selection of carbon black can lead to sufficiently non-conductive black compounds. According to Reference 6, resistivity is dependent on carbon black characteristics which include particle size, density, degree of aggregation and agglomeration, and the presence of surface oxides. In noncrystalline polymers changing resistivity with temperature can result from deagglomeration of the particles induced by increased Brownian motion caused by reduced viscosity and increased segmental motion of polymeric molecules. In crystalline polymers changes with temperature result from volume expansion of the polymer as it melts, and creation of a single melt phase.

A limited number of high-resistivity compounds were formulated and evaluated. As indicated above the resistivity characteristics were evaluated at room and elevated temperatures. In addition, characterization of physical properties also occurred as shown in Table 4-I. These

Table 4-I. Characterization Properties

Characteristic	V	CA
Ultimate Strength	RT	RT
Ultimate Elongation	RT	RT
Shore A Hardness	RT	RT
Weight Change	RT	RT
Volume Change	RT	RT
Resistivity	RT, 91, 240, 288C	RT, 91, 240, 288C

measurements were made on both virgin (V) specimens and specimens chemically aged (CA) in a synthetic geothermal brine with the following constituency: 330 ppm H₂S, 1000 ppm CO₂, 25,000 ppm NaCl, and balance H₂O. The candidates were immersed in the synthetic brine for 22 hours at 260C (500F). This brine was developed under the original DOE/L'Garde elastomer

development program.¹ Also as shown in Table 4-1 are the temperatures at which each property was measured. Note that resistivity was measured at a series of elevated temperatures, nominally 91C (195F), 204C (400F), and 288C (550F).

4.3 DEVELOPMENT

This subsection reports the compound development and evaluation that occurred.

4.3.1 Formulation Effort

The Y267 EPDM has a high level of SAF black which is a very small particle size black which provides the excellent extrusion resistance which was needed for packer seals, but provides the wrong effect insofar as electrical resistivity is concerned. This effort holds the Y267 EPDM formulation constant except for the investigation of the filler system with the objective of maintaining the excellent hydrothermochemical properties while increasing the electrical resistivity. Both black and nonblack fillers were investigated.

Table 4-II presents the scope of formulations investigated. Each of the fillers is briefly discussed below.

SAF, FEF, and MT blacks are common carbon blacks used in the elastomer industry. Super Abrasion Furnace (SAF) and Fast Extrusion Furnace (FEF) as their names imply are manufactured by burning oil in furnaces. SAF is very small particle size and FEF low medium, are respectively 20 millimicron and 40 millimicron. The ASTM designations are N110 and N550 respectively. Medium Thermal (MT) is manufactured by impinging natural gas onto hot brick which produces large particle size black, 300 millimicron. This is a common black used as a diluent to reduce the cost of compounds. The ASTM designation for MT black is N990.

Austin Black® is bituminous coal fines produced by Slab Fork Coal Company. 99% of its particles are smaller than 45 microns. It is primarily used as a cost reducing diluent, however, there is evidence that Austin Black® enhances the hydrothermochemical stability of Viton® compounds.¹

TABLE 4-II. GEOTHERMAL ELECTRICAL EPDM CANDIDATE FORMULATIONS

CONSTITUENT	Y267	312	313	314	315	316	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334
Norde1 1660®	100 ^a	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Cyanox 2246®	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
DiCup R®	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Hypalon 20®	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Antimony Trioxide	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Hyst1 B3000®	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20
SAF	75	10	25																		
FEF				25	50	75						30	40	40	40	40	40	40	40	40	
MT							25	50	75												
Austin										50											
Aerosil R972®											10										20
Mistron ZSC®														25							
C400															15	25	35				
#1 Barytes																		40	70	100	

CONSTITUENT	335	336	337	338	339	340	341	342	343	344	345	353	354	355	356	357	358	359	360	361	362
Norde1 1660®	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100	100
Cyano 2246®	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5	0.5
DiCup R®	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5	3.5
Hypalon 20®	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Antimony Trioxide	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5	5
Hyst1 B3000®	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20	20
FEF																					10
Austin										60	75										
Aerosil R972®	30																				
Hisi1 215®		20	30																		
Burgess KE®				75	125	175															75
#1 Barytes																					
Translink 37®							75	125	175							50	100	200			
C400																			50	100	150
Cyprubond®												50	100	150							

a) All amounts are in phr or parts per hundred rubber by weight, e.g. 100 phr ZnO means an amount equal in weight to the elastomer.

Aerosil R972® and HiSil 215® are very fine particle size silica reinforcing fillers in the 20 millimicron range. The R972 is a high-purity, highly-dispersed amorphous silica especially treated to provide hydrophobic properties and is manufactured by Degussa. The 215 is a compressed powder amorphous silica manufactured by PPG Industries.

Mistron ZSC®, C400, and Mistron Cyprubond® are platey talcs produced by Cyprus Industrial Minerals. 90% of the particles for each are smaller than 5.5, 18, and 4.5 microns respectively. Pure talc or magnesium silicate consists of 63.5% SiO₂, 31.7% MgO, and 4.8% H₂O. The platey structure sets up a more tortuous path in front of diffusing particles including electrical charges, thus providing good resistivity and impermeability. Talc is inherently hydrophobic and ZSC has been surface treated with zinc stearate to make it more so. C400 is a "workhorse" talc and Cyprubond® has been treated with an organofunctional silane to make it more reactive with the elastomer.

Burgess KE® and Translink 37® are surface treated calcined clays from the Southern U.S. They are surface treated probably with Silane for better reactivity and calcined to remove the water of hydration. The clays are aluminum silicates that are approximately 44% alumina and 52% silica. The clay has a platey structure which accounts for its effect on modulus and hardness. Most of the particles are in the 0.1 to 1.0 micron range but with a significant fraction in the 2 to 5 micron range. The KE® is supplied by Burgess Pigment Company and the Translink® by Freeport Kaolin Company.

No. 1 Barytes is a natural barium sulfate from the Southern U.S. It is ground into particles ranging from 2 to 25 micron with 80% under 15 micron which is the average particle size. Its chemical stability makes it an ideal pigment. No. 1 Barytes is distributed by C.P. Hall.

4.3.2 Experimental Results

As described in subsection 4.2, a set of physical characteristics and room temperature resistivity were measured for all the candidate formulations in their virgin state and after chemical ageing in brine. Then selected compound resistivities are measured at elevated temperature.

The primary objective of this task was to develop high resistivity compounds while still maintaining the good physical properties and hydrothermochemical characteristics of Y267 EPDM. Forty-two different formulations incorporating fourteen different filler systems were compounded. All formulations were not evaluated because of an associated processing problem which is discussed below.

In general all measurements were made on virgin specimens and specimens that were aged in synthetic geothermal brine for 22 hours at 260C (500F). The tensile measurements were made using ASTM D412 Die C specimens. Elongation was measured at the grips and the measurements were run at room temperature. In the majority of cases 3 specimens were pulled, but in some instances because of material limitations only 2 were pulled. Shore A hardness was measured on the double thick tensile specimen, 3 separate measurements on each of the 3 or 2 separate specimens. Resistivities were measured using ASTM test sheets according to L'Garde test procedure, 10499, contained in Appendix A. Weight and volume retention after brine ageing were measured on the tensile specimens. The volume was measured by weighing the specimens dry and immersed in water.

Table 4-III summarizes the characteristics measured on the 42 compounds. In the case of the mineral fillers; Mistron ZSC[®], HiSil 215[®], Translink 37[®], Cyprubond[®], C400, and the Burgess KE[®]; there was a processing problem. In molding the ASTM slabs blistering occurred resulting in a defective slabs. The Burgess KE[®] aluminum silicate clay was most baffling because there were no problems with the first moldings but after a little over three months and thereafter there were problems. A significant effort was spent attempting to solve the problem, but the cause or solution was not found within the restrictions of the budget. This processing problem was experienced on all compounds in Table 4-III where the column is marked NG.

Various black fillers were evaluated to determine the resistivity sensitivity to black type and amount. The results were pretty much as anticipated, however, the quantitative data provided invaluable compounding guidance. The SAF black (312, 313, and Y267) appears to trend below 10^{11} ohm-cm at about 30 phr (see footnote on Table 4-II for definition of phr). FEF and MT black (314, 315, 316 and 320, 321, 322) behave similarly and pass through 10^{11} ohm-cm at about 40 phr. Austin Black (323,344,345)[®], bituminous coal fines, shows good resistivity at the highest level investigated, 75 phr. However, Austin Black[®] does not render reinforcing properties to the elastomer. MT black, though better than Austin Black[®], reinforces the compound somewhat but not nearly as well as FEF black which provides the same resistivity characteristics.

TABLE 4-III. COMPOUND CHARACTERISTICS

CHARACTERISTIC	Y267	312	313	314	315	316	320	321	322	323	324	325	326	327	328	329	330	331	332	333	334
Ultimate Tensile Strength, psi V	1629	991	1178	1713	1803	1940	1286	1465	1563	1172	907	1277	1516	NG	1755	1422	1348	1850	1448	1564	694
psi CA	b	1102	1378	1586	1844	1881	1311	1592	1675	1154	961	1407	1562		1936	1513	1252	1779	1491	1580	804
% CA/V	b	111	117	93	102	97	102	109	107	98	106	110	103		110	106	93	96	103	101	116
Ultimate Elongation, % V	170	278	237	249	197	148	252	244	175	180	388	191	182		196	167	175	206	225	218	230
% CA	b	282	247	222	210	146	246	228	181	174	362	187	172		208	181	188	196	201	209	320
% CA/V	b	101	104	89	107	99	98	93	103	97	93	98	95		106	109	107	95	89	96	139
Shore A Hardness V	90	66	74	75	83	89	75	83	86	81	67	79	81		82	83	84	83	84	84	76
CA	b	66	73	75	81	87	74	82	87	81	67	78	81		83	84	82	82	83	83	75
CA/V	b	100	99	100	98	98	99	99	101	100	100	99	100		101	101	98	99	99	99	99
Resistivity ohm cm V	<4E8	2E14	5E12	2E14	5E9	<4E8	2E14	<4E8	<4E8	2E14	2E14	2E14	3E11		2E73	3E12	9E12	6E11	5E11	6E11	2E14
ohm cm CA	b	3E14	2E14	3E14	<4E8	<4E8	4E14	1E10	8E9	6E14	4E14	4E14	2E10		2E13	2E13	3E13	2E11	9E10	1E11	1E14
% CA/V	b	150	4000	150	10	100	200	2500	2000	300	200	200	7		100	667	333	33	18	17	50
Weight Retained % CA/V	b	99.9	100.7	101.0	101.5	102.1	100.2	100.5	100.3	99.8	100.0	101.2	101.1		102.5	102.9		101.2	101.2	101.1	99.3
Volume Retained % CA/V	b	100.1	99.7	100.2	100.6	100.7	99.3	100.3	100.3	99.4	100.1	100.5	100.0		101.9	101.7		100.8	101.0	101.2	98.6

CHARACTERISTIC	335	336	337	338	339	340	341	342	343	344	345	353	354	355	356	357	358	359	360	361	362
Ultimate Tensile Strength, psi V	884	NG	NG	1233	1208	1412	NG	NG	NG	1174	1193	NG	NG	1361 ^a	789	750	831	NG	NG	NG	NG
psi CA	1091			1352	1319	1431				1102	1176										
% CA/V	123			110	109	101				94	99										
Ultimate Elongation % V	297			243	171	193			139	180			186	222	211	241					
% CA	375			261	205	206				141	139										
% CA/V	126			107	120	107				101	77										
Shore A Hardness V	82			79	84	85				85	88			86	71	72	74				
CA	80			80	84	84				85	86										
% CA/V	98			101	100	99				100	98										
Resistivity ohm cm V	2E14			2E14	2E14	2E14				2E14	3E13				>9E14	>9E14	>9E14				
ohm cm CA	1E14			2E14	1E13	3E12				2E14	2E14										
% CA/V	50			100	5	2				100	667										
Weight Retained % CA/V	99.7			100.6	100.8	101.1				101.4	101.1										
Volume Retained % CA/V	99.7			100.1	100.0	100.4				100.9	100.6										

a) One measurement, slab had numerous bubbles.

b) No measurements, this ageing typically results in virtually no change.

Again because of budget constraints, four compounds were selected for resistivity measurements at elevated temperature. These were relatively complex tests which were carried out in a nitrogen atmosphere oven. The specimens were first immersed in room temperature water for a minimum of 40 hrs. They were washed with distilled water, blown dry, and the resistivity measured. They were then simultaneously air dried in the open room for a minimum of 40 hours. and the resistivity measured. The specimens were placed in the 91C (195F) for about 18 hours and the resistivity measured. The specimens were then equilibrated at 204C (400F) for 1.5 hr. after which the resistivity was measured. The specimens were then equilibrated overnight for 18 hrs. at 288C (500F) after which the resistivity was measured again. The specimens were then cooled below 149C (300F) in nitrogen and then cooling was finished in the room. The final room temperature resistivities were measured.

Table 4-IV summarizes the series of measurements which include the

Table 4-IV. Resistivities At Temperature and Other Conditions

Condition	313		323		328		338	
	V ohm-cm	CA ohm-cm	V ohm-cm	CA ohm-cm	V ohm-cm	CA ohm-cm	V ohm-cm	CA ohm-cm
Wet Soak 40 hr.	7E12	4E9	4E10	4E10	2E13	1E13	1E14	4E9
Air Dry 40 hr.	4E9	5E14	7E14	3E9	4E9	3E9	9E14	4E9
Temperature								
91C (195F)	4E13	2E14	2E14	2E14	2E13	3E13	2E14	9E10
204C (400F)	4E9	1E14	3E12	2E13	3E13	4E10	3E12	3E14
288C (550F)	2E12	2E12	1E11	1E12	2E12	1E9	4E11	2E13
RT	5E9	3E9	7E14	4E14	<4E8	<4E8	>9E14	>9E14

high-temperature exposure. The four compounds selected for these measurements are 313 (25 phr SAF), 323 (50 phr Austin[®]), 328 (40 phr FEF/15 phr C400) and 338 (75 phr Burgess KE[®]). All four survived the gauntlet of test conditions without the resistivity falling below 10^9 ohm-cm, except 328. On the last measurement at room temperature after surviving all the previous conditions, the resistivity of 328 fell below 4×10^8 ohm-cm. For 313, 323, and 338 the general trend is reducing resistivity with temperature. However, the 313 chemically aged specimen experienced erratic behavior, and both the virgin and chemically aged specimens reached their

behavior, and both the virgin and chemically aged specimens reached their minimums at the end of the test series where they remained. Except for the wet soak points and the chemically aged point prior to oven drying, 323 experienced a well behaved reversible trend with temperature measuring between 10^{11} to 10^{15} ohm-cm. 338 behaved similarly to 323 except apparently the chemically aged specimen took a little longer to dry out and move with the general trend. 328 shows an erratic behavior and based on the limited data it is premature to try to postulate any trends.

4.4 CONCLUSIONS REGARDING INSULATIVE COMPOUNDS

The limited development effort yielded three compounds with good resistivity characteristics which were selected for the final phase of this effort, 323 (50 phr Austin®), 331 (40 phr FEF/40 phr Barytes), and 338 (75 phr Burgess KE®). The Austin Black® and Burgess KE® provide very good resistivity throughout the various conditions tested while the FEF black (331) provides very good physical properties. 331 was revisited even though it was not carried into resistivity at temperature tests because of the blistering during molding problems experienced with a similar compound, 328 (40 phr FEF/15 phr C400), discussed above in Subsection 4.3.

Ultimately the field was narrowed to 323 and 331 because 338 also suffered from the blistering problem and was abandoned after several unsuccessful attempts were made to identify the problem cause. It would have been desirable to further optimize some of the compounds and to identify the source of and solution to the blistering since 338 was an attractive compound otherwise, however, the budget did not permit. 323 has good electrical properties. Its resistivity over the full range of testing measured between 3×10^9 and 7×10^{14} ohm-cm, however, its strength is marginal. As discussed above 331 was revisited and its primary attraction is the higher strength afforded by the FEF black. It was not tested at elevated temperatures, but it was tested at room temperature. The virgin specimen measured 6×10^{11} ohm-cm and the chemically aged 2×10^{11} ohm-cm. At temperature it is expected to have similar properties to 338, another FEF black compound which had a resistivity range from 4×10^9 ohm-cm to 1×10^{15} ohm-cm.

As a conclusion to this task, connector boots were made from 323 and 331 and provided to Los Alamos National Laboratory (LANL) for testing. LANL intended to install the connector boots in an actual cablehead, immerse it in an autoclave under temperature and pressure, and measure the conductor-to-conductor resistance. The new compounds were to be simultaneously run with and compared against the currently used commercial

compound. The following suggested resistance measurements were provided with the boots:

- a) Room temperature, dry
- b) Room temperature, wet only if test intentionally exposes boots directly to water. If this is the case, resistance versus time exposed will be pertinent.
- c) As a function of temperature (every 50F) during the autoclave heating. Polymers typically change resistivity by a few orders of magnitude over the temperature range of interest. If the test would normally expose the boots to air or oxygen, suggest that the first test be in nitrogen in case the oxygen causes pertinent changes.
- d) As a function of time while at test temperature.
- e) As a function of temperature (every 50F) during cooling to detect any irreversibility.
- f) As a function of time (morning and afternoon) in ambient room for about 3 days to detect reversibility.

Results of the testing at LANL were not available prior to the completion of this project.

5.0 GEYSERS EXPLOSIVE STIMULATION EXPERIMENT SEALS

This section discusses L'Garde's support of the first Explosive Stimulation Experiment carried out in a geothermal well.

5.1 BACKGROUND

Union Oil, Physics International, and Rocket Research engaged in a project with the DOE through Los Alamos National Laboratory (LANL) to explosively stimulate a geothermal well at the Geysers.⁹ This was the first known attempt to stimulate a geothermal well using explosives. Union Oil developed the overall enveloping canister vessel and provided the well in which the experiment was run. Physics International developed the detonator package and Rocket Research developed the nitrate salt explosive, Hitex II. The objective of the experiment was to demonstrate that a large explosive package could be safely and successfully emplaced and detonated in a geothermal well. The objective was successfully demonstrated.

5.2 NEED FOR Y267 EPDM

Development tests were run on the detonator package in the LANL autoclave at 260-275C (500-525F). Problems existed with both the Teflon[®] and Viton[®] vessel seals which needed to perform for 48 hours. The Teflon[®] seals were extruding to the extent of disappearing and the Viton seals were embrittling.

After several alterations and modifications, a system evolved which sealed. However, there was still concern as to the reliability of the sealing system so L'Garde was contacted regarding the Y267 EPDM technology.

Based on numerous previous tests, it was clear without any further testing that for the autoclave environment, i.e., steam/water, the DOE/L'Garde Y267 EPDM was the best candidate. However, the seals may also be exposed to the explosive in that the detonator package is immersed in the explosive. Consequently compatibility of the Y267 EPDM with the explosive was a significant question.

Fortunately at about this time, tests were being planned to confirm the compatibility of various components in the nitrate salts explosive.

L'Garde recommended that several elastomer compounds be piggybacked to this test. The recommendation was adopted and L'Garde provided specimens from the following compounds developed under L'Garde's DOE GEM elastomer development program¹:

- 255 - Viton® (VTR-4590)
- 266 - EPDM/Viton® blend
- Y267 - EPDM

These three plus the Viton® compound that was currently being used were tested in the explosive.

The test was run at Physics International and the three L'Garde compounds were placed in 3/8 inch ID vials. Two to three tensile specimens were placed in each vial with one compound per vial. The specimens were trimmed at the grips to narrow them to 5/16 inch so that they would fit into the vials. The test was run for 24 hours at 243-246C (470-475F). There was a 6-7 hour heat-up, and cooldown occurred over several hours. The autoclave was pressurized with nitrogen at about 6.7 MPa (1000 psi) at temperature.

The results of this test indicated that of the four elastomers tested, Y267 EPDM was the only viable candidate. The results are shown in Table 5-I and it can be seen that the elongation at break were very low for

Table 5-I. Results of Immersion Tests In Explosive

COMPOUND PROPERTY	255 Viton®	266 EPDM/Viton®	Y267 EPDM
Tensile Strength, psi			
Original, psi	2244	1420	1962
Explosive Aged, psi	492	1479	1084
% Retained	21.9	104.2	55.2
Elongation @ Break, %			
Original	303	276	278
Explosive Aged	42	33	109
% Retained	13.9	12.0	39.2

both the 255 and 266 compounds. The Viton® O-ring indicated similar results also. Based on these tests the Y267 EPDM was clearly the best elastomer for sealing the explosive. There was already a wealth of data which indicated that the Y267 EPDM was the best choice in the geothermal steam environment.

5.3 Y267 EPDM SEALS PROVIDED

Based on the autoclave tests it was decided to use Y267 EPDM seals on the detonator vessel, the outer containment canister, as well as the release device that released the entire explosive device once it was located in the well.

Given the go-ahead the schedule required that molds and the Y267 EPDM seals be made in 5 weeks. Eight each of the following O-ring seal sizes were delivered to Union Oil:

- Release Device --
 - 2-009
 - 2-118
 - 2-119
 - 2-126
- Detonator Vessel --
 - 2-153
 - 2-155
- Outer Containment Canister --
 - 2-438

5.4 EXPERIMENT RESULTS

Two field tests were run; a subscale preliminary test to check out the system, and then the final full-scale test. The subscale test was run on 4 December 1981 with 367 kg of explosive and the full scale test was run on 14 December 1981 with 5000 kg of explosive. In both cases the detonation was set to occur 48 hours after timer initiation and did occur as scheduled. The fact that the detonations occurred precisely on schedule and explosions were nominal, imply that the seals performed successfully.

The overall experiment successfully demonstrated that a large explosive device can be safely emplaced and successfully detonated in a hot geothermal well without causing damage to the well bore or casing.

6.0 BRIEF MINISUPPORT TASKS

This section contains the balance of the effort performed on the GEM-TA Program.

6.1 OPERATOR BRIEFINGS

One of the more major roadblocks to the adoption of new technology into the drilling sector of the geothermal, and oil and gas industry arises because of the continual urgent atmosphere which prevails. When jobs are carried out on a priority basis, the number of steps or items of hardware which require special attention must be minimized to accomplish the schedule objective.

This omnipresent urgent atmosphere especially affects the introduction of new elastomer technology because it is a relatively long lead-time item and any changes usually result in problems. Furthermore, many of the rubber seals are special shapes, hence, special molds exist at the preferred molded products vendor who is the usual supplier for the particular part and chances are he will probably not be familiar with the specific new technology of interest. Even with standard parts such as O-rings, because of the numerous sizes and elastomer compounds, except for the most common, stocking is infeasible so parts made from a new compound will probably still be a special order taking 6 to 8 weeks.

Consequently because elastomeric parts made from a new compound imply a significant effort, long-range planning, and vulnerability to problems, massive inertia exists resisting adoption of new elastomers into the industry.

It was reasoned that with limited funding to challenge this inertia, the greatest impact for the dollar should result by informing the ultimate benefactor. He directly benefits from the advances and is not hampered by non-technical biases against their use such as logistics and uncertainty of the unknown. A fairly long chain of organizations exist which lead up to the ultimate benefactor which can include part of all of the following: rubber mixer, molded parts manufacturer, tool manufacturer, service company, and the operator. By informing the operator of the capabilities of the Y267 EPDM, he would then be aware of the potential cost savings and extended capabilities the new technology will afford. If he

becomes motivated to specifically specify Y267 EPDM components in his orders for equipment and services, he would thus force the entire chain of organizations to respond to a ripple down or domino fashion.

To this end direct briefings describing the history of the GEM elastomer compound development, and the performance of Y267 EPDM were offered to operators and other pertinent organizations in the following regions:

- Los Angeles Area
- San Francisco Area
- Santa Rosa
- Salt Lake City
- Dallas
- Houston

Table 6-I gives the organizations who were briefed and the primary point of contact.

Table 6-I. Organizations Briefed

<u>Region</u>	<u>Organization</u>	<u>Point-of-Contact</u>
Los Angeles	Magma Power	E. Zajac
	MCO	R. Russell
	Occidental Research	P. LaMori
	Republic Geothermal	T. Turner
	Union Oil	D. Pyle
San Francisco	Chevron	S. Garrett
	GRI	J. Combs
	Thermal Power	W. D'Olier
	U.S. Geological Survey	P. Muffler
Santa Rosa	Aminoil	W. House
	California Energy	W. Tipton
	Union Oil	D. Ash
	Thermogenics	J. Argenti
Salt Lake City	Phillips	T. Turner
Dallas	Arco	J. Hellinghausen
	Hunt Energy	W. Rodgers
	Sun Oil	E. Western
Houston	Battelle	M. Watkins

6.2 Cementing Wiper Plug

The Hot Dry Rock (HDR) Geothermal Project¹⁰ current activities center around a reservoir system of two wells drilled to about 4.6 km (15,000 ft.) and completed at an angle of 35° from vertical. Communicating fractures must be created which allow fluid to flow from one well to the other. The fractures are planned to be created hydraulically using a cemented-in liner/PBR as one method. A whole host of potential problems existed because the cementing, though not at the bottomhole temperature of 320C (608F), still occurred at a very high temperature.

One of the many problems is maintaining the functional integrity of the elastomeric cementing wiper plug. The wiper provides a moving barrier or separation-plug which follows the cement into the well and isolates the cement from contamination. The wiper is constructed primarily of rubber with a series of circumferential wiping ribs as shown in Figure 6-1. The integrity of the elastomeric wiper throughout the cementing process is of utmost importance to maintain the purity of the cement which is required for a good cement job.

In anticipation of this requirement, Los Alamos National Laboratory (LANL) had run autoclave tests on various candidate wiper plugs none of which withstood the 280C (536F) test temperature without degradation. One of the wipers completely disintegrated and disappeared. Because of this problem and their awareness of the performance capability of the Y267 EPDM, LANL determined that they needed Y267 EPDM cementing wiper plugs.

After months of requesting Y267 EPDM wiper plugs from at least two cementing contractors, Dowell agreed to provide them to LANL. "Y267 EPDM" implies a specific recipe and process for making elastomeric parts. Because some Y267 components are not readily available and the process requires post-curing at 288C (550F) in a nitrogen atmosphere, Dowell obtained the uncured Y267 EPDM from L'Garde, molded the cementing wiper plugs, had L'Garde post-cure them, and then L'Garde returned the plugs to Dowell.

Once LANL took possession of the wiper plugs they immersion tested them in the autoclave at 280C (536F) and 17.2 MPA (2500 psi). The heat-up occurred over a period of somewhat over 3 hours. The temperature was held for 30 minutes and then cooled down over a period of about 20 hours. They recorded changes in appearance and hardness. The hardness was measured at 10 different points on the wiper with most in the range of 0 to 3 Shore A points lower after exposure. Because of the difficulty of making repeatable hardness measurements even under more ideal conditions and

especially on this rounded geometry, much of the differences were attributed to measurement error. Figure 6-1 shows the Y267 EPDM wiper after immersion testing.¹⁰

At this writing, one Y267 EPDM Dowell cementing wiper plug was run on the HDR project at Fenton Hill. A "scab" liner was cemented in at about 4389M (14,400 ft.) where the BHCT temperature was 275C (525F). The cement job was successful, however, during drill-out the cement plug was not located and hard cement was tagged 26M (70 ft.) below the wiper catcher. Therefore, this field use is termed inconclusive.

6.3 SPONTANEOUS TECHNOLOGY TRANSFER

The path from conclusion of small development efforts to practical application of the technology is clouded with uncertainty. In many cases the situation is one of chicken and egg where if someone isn't available to service inquiries no demand is created, and without a demand manufacturers cannot justify setting-up to service inquiries.

This dilemma was avoided with the Y267 EPDM elastomer because the DOE foresaw the need and provided the mechanism for servicing inquiries about the technology and primarily through the GEM Technology Transfer Contract funded through Brookhaven National Laboratory¹¹, and to a limited extent through this contract. Much of the success in the transfer of the Y267 EPDM technology can probably be attributed to the transfer effort which breaks the technology out of the chicken/egg cycle. This subsection summarizes the spontaneous technology transfer which occurred under the GEM-TA Program in response to inquiries, as opposed to efforts initiated by L'Garde.

Figure 6-1. Dowell Y267 EPDM Cementing Wiper Plug After 280C (536F) Exposure. Note As-New Condition.

Numerous telephone, letter, and first person inquiries were satisfied by providing instant information, by mailing written information, and by providing samples. The written information included papers and more specific information requested and available.

The inquirers included over 100 organizations and many of them made multiple contacts. They included the following types of organizations:

- Other DOE projects
- Department of Defense Organizations
- U.S. Government Laboratories
- Drilling Measurements Companies
- Inline Chemistry Instrumentation Companies
- Wellhead Manufacturers
- Rubber Chemicals Manufacturers
- Packer Manufacturers
- Molded Products Manufacturers
- Electric Utilities
- Oil Field Equipment R&D Centers
- Pipeline Pump Manufacturers
- Plate Heat Exchanger Manufacturers
- Downhole and Surface Pump Manufacturers
- Logging Tool Companies
- Oil Company R&D Centers
- Oil Field Service Companies
- A&E Contractors
- Drilling Technology Consultants
- Oil Field Equipment Manufacturers
- Polymer Manufacturers
- Logging Companies
- University Research Centers
- Atomic Energy R&D Centers
- Turbine Manufacturers
- Nuclear Electrical Cable Manufacturers

Most of the inquiries were American but some were foreign which include the following countries:

- Israel
- France
- Japan
- Sweden
- England
- Canada

6.4 PRESENTATION OF PAPERS AT TECHNICAL SYMPOSIA

Presentation of technical papers always provides a highly efficient means of disseminating and transferring technology. For a given effort, a broad audience can be addressed directly at the conference, and then indirectly through the research literature when the meeting transactions or the papers become available to the community. The papers may also be subsequently published in technical journals providing significant additional exposure.

During the course of the GEM-TA Program three presentations of the technology were made at various symposia. The reference information regarding each is listed in Table 6-II and the papers are included as Appendices B through D.

TABLE 6-II. GEM-TA PAPERS

Date	Conference	Location	Title
October 1981	GRC Annual Meeting	Houston, TX	Geothermal Packers and Packer Elastomers
December 1981	High-Temperature Electronics and Instrumentation Conference 1981	Houston, TX	High-Temperature Y267 EPDM Elastomer Field and Laboratory Experiences, August 1981
February 1983	IADC/SPE 1983 Drilling Conference	New Orleans, LA	A Proven Elastomer Compound for Extremely Hostile Geothermal and Oilfield Environments

7.0 REFERENCES

1. A. R. Hirasuna, et. al., "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976-30 June 1979," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy-Division of Geothermal Energy, July 1979.
2. A. R. Hirasuna, et. al., "A Proven Elastomer Compound for Extremely Hostile Geothermal and Oilfield Environments," IADC/SPE 1983 Drilling Conference, 20-23 February 1983, New Orleans, LA
3. A. R. Hirasuna, "Application of Geothermal Elastomer Development to Hot Dry Rock Downhole Tools," L'Garde Final Report No. LTR-80-AH-101, December 1980.
4. A. R. Hirasuna, "Application of Rubber Bearings For Geothermal Lineshaft Pumps," Information Release 235, 3 August 1982, L'Garde, Inc., Newport Beach, CA 92663.
5. G. J. Friese, "Short and Long-Term Tests of Elastomers With Hot Hostile Fluids," L'Garde, Inc., Newport Beach, CA 92663, Brookhaven National Laboratory/U.S. Department of Energy, Division of Geothermal Energy, Report No. LTR-82-GF-110, 30 December 1982.
6. Andries Voet, "Temperature Effect of Electrical Resistivity of Carbon Black Filled Polymers," Rubber Chemistry and Technology, Vol. 54, p.42, Mar-Apr 1981, Rubber Division American Chemical Society.
7. Letter from Alfred Hess, U. S. Geological Survey, to Alan R. Hirasuna dated November 1980.
8. Letter from Charles Arnold, Jr., Sandia National Laboratories, to Alan R. Hirasuna dated 3 December 1980.
9. Douglas M. Mumma, et. al., "Geofrac -- An Explosive Stimulation Technique for A Geothermal Well," Geothermal Resources Council 1982 Annual Meeting, San Diego, CA, 11-14 October 1982.
10. Roland Pettitt, et. al., "Downhole Cement Test In a Very Hot Hole," Geothermal Resources Council 1982 Annual Meeting, San Diego, CA, 11-14 October 1982.
11. A. R. Hirasuna, et. al., "Geothermal Elastomeric Materials Technology Transfer (GEM-TT) Program, Final Report," L'Garde, Inc. Report No. LTR-82-AH-111, December 1982.

APPENDICES

REV
SH
DWG. NO.

APPLICATION

REVISIONS

NEXT ASSY	USED ON	REV.	DESCRIPTION	DATE	APPROVED
-----------	---------	------	-------------	------	----------

APPENDIX A

UNLESS OTHERWISE SPECIFIED
DIMENSIONS ARE IN INCHES
TOLERANCES ARE:

FRACTIONS DECIMALS ANGLES
± .XX ± ±
XXX ±

MATERIAL

FINISH

DO NOT SCALE DRAWING

CONTRACT NO.

APPROVALS

DATE

DRAWN

G. Friese

6/18/82

CHECKED

ISSUED

L'GARDE, INC.

1555 PLACENTIA AVENUE
NEWPORT BEACH
CALIFORNIA 92663

DC VOLUME RESISTIVITY OF INSULATING MATERIALS
(ASTM D257-78)

SIZE
A

FSCM NO.

DWG. NO.

10499

REV.

SCALE

SHEET

DC VOLUME RESISTIVITY OF INSULATING MATERIALS

(ASTM D257-78)

This specification provides the detailed procedure for determining the volume resistivity of insulating materials. The test specimen must be flat and at least 5 inches in diameter.

1.0 CONDITIONING

Test specimens and test electrodes shall be maintained at $73.4\text{ F} \pm 3.6\text{ F}$ ($23\text{C} \pm 2\text{C}$) temperature and at $50\% \pm 5\%$ humidity for at least 40 hours prior to testing, and at such temperature and humidity during test.

1.1 SULFURIC ACID SOLUTION

Approximately a half-inch of solution is required in the conditioning chamber (ice chests) dish. If a clean solution is available, go to Step 1.1.3.

1.1.1 Add 70 ml of concentrated sulfuric acid to 150 ml of purified water. (CAUTION: Never add water to concentrated sulfuric acid; it will explosively boil.)

1.1.2 Cap or cover and cool to $75\text{F} \pm 10\text{F}$.

1.1.3 Place solution into a clean, tall beaker.

1.1.4 Read and record temperature and specific gravity. $T_s = \underline{\hspace{2cm}}$ of
 $SG_T = \underline{\hspace{2cm}}$.

1.1.5 Enter chart entitled "Sulfuric Acid Solution Requirements" with these two values. If the point is within the shaded area, the solution is satisfactory.

SULFURIC ACID SOLUTION REQUIREMENT

- 1.1.6 If outside the shaded area, bring it into the shaded area by adding purified water or sulfuric acid as indicated on the chart. (After addition of either fluid; stir, read both temperature and specific gravity, and re-enter the chart with the new values.) Record final values. $T_s = \text{_____}^{\circ}\text{F}$
 $SG_T = \text{_____}$.
- 1.1.7 Pour in chamber dish. Repeat above until enough solution has been made or adjusted to have about $\frac{1}{2}$ inch depth of solution.
- 1.1.8 CAUTION: Do not leave chamber open unnecessarily.
- 1.2 Cut a hole in the edge of each test slab to be conditioned.
- 1.3 Clean slab with MEK. (CAUTION: Touch only the edges of the slabs after cleaning until resistivity is measured.)
- 1.4 Hang on hooks in humidity chamber for 40 hours minimum. If a test piece is thicker than 0.28 inch (7mm), condition for at least 88 hours.
- 2.0 RESISTIVITY TEST SET-UP AND CALIBRATION
- 2.1 Fill the dish in the test chamber with about $\frac{1}{2}$ " depth of sulfuric acid solution. Make or correct this solution per Step 1.1.
- 2.2 Plug line cord into a 115 volt, 60 CPS, 1 phase, three-prong receptable.
- 2.3 Turn the range selector switch to the DISCH position.
- 2.4 Turn ADJ. A control clockwise to turn the vibrotest mehoammeter Model 2851. Allow a warm-up time of approximately 2 minutes.

- 2.5 Turn 100/500 volt switch to 500 volt position.
- 2.6 Turn ADJ. A control so that meter pointer lines up with ∞ at the counter-clockwise end of scale. (During checkout, it was found that this couldn't be done; in such a case, rotate ADJ.A fully counterclockwise without shutting off machine.)
- 2.7 Turn selector switch to CHK. B position. The meter pointer will deflect toward the clockwise or "B" end of the scale.
- 2.8 Turn ADJ. B control until meter pointer lines up with the 1 megohm point at the clockwise end of the scale.
- 2.9 Switch the VOLTS switch to 100 volts and switch the GROUND switch to GRD. Make sure the output leads are not shorted together.
- 2.10 Rotate the range selector switch to the X100K position.
- 2.11 Adjust the large knob concentric with the ADJ A control so that the meter pointer indicates ∞ at the A end of the scale.
- 3.0 TESTING
- CAUTION: Always turn the range selector switch to the DISCH position when handling the leads.
- 3.1 Turn range selector switch to DISCH, volts switch to 500 VOLTS, and ground switch to RET.
- 3.2 Just before performing a series of tests, read and record the temperature and humidity in both the conditioning and test chambers. If outside of requirements, notify test engineer. If there is no method of directly reading humidity, so state on data sheet -- unless atmospheric conditions have been very dry (Santana winds) or very wet (raining); in this case, determine humidity per the Appendix.

Remove the guard and guarded electrodes from the test chamber and place upside down on table to protect surfaces.

- 3.3 Clean with MEK the resistance test equipment surfaces that will touch the rubber.
- 3.4 Remove test specimen from conditioning chamber (handling by edges only) and place on unguarded (square) electrode in test chamber.
- 3.5 Put guarded electrode (cylinder) on top of specimen and push down hard. Add guard electrode (tube) such that the gap between it and the guarded electrode is constant to the eyeball.
- 3.6 Connect test lead to the cylinder, the return lead to the square and the guard lead to the tube.
- 3.7 Close test chamber door. (Close the conditioning chamber door also if you hadn't already.)
- 3.8 Turn the range switch to X1.
 - 3.8.1 Overload light comes on: Turn the switch back to DISCH, mark the resistance column of the data sheet "OL", and skip to Step 3.11.
 - 3.8.2 Needle goes to the right (off scale) of 1, wait one minute in case it returns. If it doesn't, record < 1 in the data sheet's resistance column, and skip to Step 3.11.
 - 3.8.3 Needle goes between 1 and 8: Wait one minute, record the value and skip to Step 3.11.
 - 3.8.4 Needle is to the left of 10: Go to Step 3.9.
- 3.9 Slowly rotate the range switch until the needle is between 1 and 10. Wait one minute. Record the values on the gauge and the range switch; for instance, "2.5" on the gauge and "X1K" on the range

switch is "2.5 X1K". Skip to Step 3.11.

3.10 If the range switch is on X100K and the gauge reads greater than 10:

3.10.1 Jiggle the range switch and wait one minute.

3.10.2 If the reading is below 20, record the gauge value plus X100K.
If the reading is above 20, record "> 20X100K".

3.11 Place the range switch on DISCH, open door, and remove the top two clips, the top two metal pieces (placing them upside down on the table), and the test specimen. Close door.

3.12 Measure the thickness of the slab in four places and record all values.

3.13 If there are more specimens to be tested, repeat Steps 3.3 through 3.12.

3.14 Turn off megohmmeter (ADJ A), and unplug.

3.15 To calculate volume resistivities:

$$\rho_v (\Omega\text{-cm}) = 35 (10)^6 R_v/t$$

where R_v is in $M\Omega$ and t is in inches. Report resistivities to one significant digit, i.e. 7 (10)¹⁷.

DC RESISTIVITY
(SPECIFICATION # 10499)

Date/Time _____ Operator _____

Pre-Test Conditions:	<u>Required</u>	<u>Conditioning Chamber</u>	<u>Test Chamber</u>
Humidity (%)	45-55	_____	_____
Temperature (°F)	70-77	_____	_____

Compound	Resistance M Ω	Thicknesses (inch) 4 Readings	Volume Resistivity (Ω -cm)

Post-Test Conditions:	<u>Required</u>	<u>Conditioning Chamber</u>	<u>Test Chamber</u>
Humidity (%)	45-55	_____	_____
Temperature (°F)	70-77	_____	_____

APPENDIX

Calculation of Chamber Humidity

1. Upon opening chamber, immediately read and record air temperature.
2. Immediately remove about 200 ml of solution and place in tall beaker.
3. Accurately measure and record solution temperature and specific gravity.

$T_A = \underline{\hspace{2cm}}^{\circ}\text{F}$

4. Calculate specific gravity at 77F to four decimal places.

$T_S = \underline{\hspace{2cm}}^{\circ}\text{F}$
 $SG_T = \underline{\hspace{2cm}}$

$$SG_{77} = SG_T + 0.0005266 (77 - T_S)$$

$SG_{77} = \underline{\hspace{2cm}}$

5. Using T_A (not T_S) and SG_{77} , enter "Humidity Control Chart" and read the humidity, interpolating to the nearest $\frac{1}{2}\%$.

Humidity = $\underline{\hspace{2cm}}\%$

A-10

HUMIDITY CONTROL CHART

APPENDIX B

GEOTHERMAL PACKERS AND PACKER ELASTOMERS

A. R. Hirasuna
C. A. Stephens

L'Garde, Inc.
Newport Beach, CA 92663

ABSTRACT

Reliability of thermal packers has been an eternal problem at temperatures beyond the 149-177C (300-350F) level. High temperature packer elastomer compounds were developed by L'Garde, Inc. for 260C (500F) environments under sponsorship of the U.S. Department of Energy in 1976 to 1979. Compounds based on four polymer systems were developed which exceed the contract requirements. The Y267 EPDM compound, particularly, has generated interest because of its good performance, economic pricing of constituents and good processability.

Several successful downhole and laboratory trials of Y267 EPDM have occurred at moderate and extremely high temperatures. Otis Engineering tested a Y267 packer seal at 302C (575F) for five days. The Y267 seal in an Otis packer was subsequently run in a Bakersfield continuous steam injection well at about 204C (400F) for 5 months. The same model packers and elements were run in Union Geothermal wells at the Baca, NM where hydraulic stimulation and diagnostics occurred at 160-171C (320-340F). Los Alamos National Laboratory tested the Y267 EPDM O-rings for a cablehead. Tests were first run in an autoclave with water and Mobil One oil to temperatures in excess of 340C (644F). The Y267 equipped cablehead was subsequently run at Fenton Hill at 317C (603F).

Further developments of packer technology are also being pursued by L'Garde. A new casing packer concept for 371C (700F) production service is now undergoing full-scale prototype development.

INTRODUCTION

In 1976, reliable packer seal elastomers for the unusually severe geothermal environment at 260C (500F) did not exist. L'Garde, Inc. developed geothermal elastomer compounds for casing packers during the period of 1976-1979 under U.S. Department of Energy (DOE) contract DE-AC03-77ET283091. The resulting developments yielded compounds from four polymer systems which successfully exceeded the contract requirements.

The development phase was previously reported at GRC conferences in 1978 in Hilo², and in 1979 in Reno³. Since that time significant downhole trials of primarily the Y267 EPDM have occurred. In addition, L'Garde is developing a new 371C

(700F) casing packer concept for the DOE. This paper presents the relatively recent downhole experience and the new packer work that is presently in progress.

ELASTOMER COMPOUND DEVELOPMENT, 1976-1979

Previous packer elastomer compound development resulted in compounds from four different polymer systems which exceeded the original contract requirements. The polymer systems include: EPDM (Nordel), FKM (Viton), EPDM/FKM Blends, and propylene/TFE (AFLAS). The packer requirements include the following environments: sour brine, 260C (500F), and 20.7-27.6 MPa (3000-4000 psi) differential pressure for 24 hours.

Though compounds from all four polymer systems exceeded the requirements, one of the compounds is particularly successful because of its good processing qualities, extremely good thermochemical stability, extremely good mechanical properties, and finally it's low cost constituents. The compound, Y267 EPDM, now has significant downhole experience and is the compound tested in the case histories given below. An additional virtue of the compound is its uncharacteristically good (for EPDM's) in hydrocarbons. Two experiences are quoted below, and another is reported in Reference 4 where at high temperatures the Y267 EPDM was the best O-ring material of 34 compounds tested from 15 different companies even in oil.

OTIS ENGINEERING CORP. PACKER TESTS

Y267 EPDM packer seals were molded by L'Garde for tests by Otis Engineering Corp.⁵ The seals are for a 7-inch casing packer which Otis tested at 302C (575F) and 20.7 MPa (3000 psi) differential pressure in their laboratory simulation tester. The elements were successfully sealing after 5 days when the test was terminated. The seal was in excellent condition considering the environment and did not show any signs of reversion softening or scission. Figure 1 shows the post mortem seal.

Prior to this test Otis tested an EPDM from another firm but at a lower temperature, 288C (550F), and the seal failed because it lost strength due to reversion softening of the polymer. The post mortem seal did not have any form and looked like a glob of tar. Otis has since tried several other competing EPDM compounds and none has yet equaled the performance of the Y267 EPDM.

Figure 1. Post Mortem Y267 EPDM Seal After 5 days of Testing @ 302C (575F) and 20.7 MPa (3000 psi). Note The Excellent Condition Considering the Extreme Severity of The Environment.

BAKERSFIELD CONTINUOUS STEAM INJECTION

The Y267 EPDM element on an R&D Otis packer was fielded in a Bakersfield heavy oil continuous steam injection well. The steam is 246C (475F) at the boiler and 204C (400F) at the wellhead.

The packer was pulled after 5 months of continuous steaming because the well required sand removal. Tracer tests confirmed that the packer was still sealing prior to removal. Typically, difficulty is encountered in retrieving thermal packers but this packer was retrieved with no problems whatsoever, where usually difficulty is encountered with the seal bonding to the casing, or hardening in place requiring the seal to be broken to enable packer retrieval.

The appearance of the recovered seal was excellent. It looked virtually as new after 5 months continuous operation and it was apparent that it could have remained in operation indefinitely. Figure 2 is a photograph of the packer seal after it was removed from the Bakersfield well. The good condition is particularly significant since the Y267 EPDM seal was in contact with crude oil which covered the packer and element when it was retrieved. EPDM's are generally considered inappropriate for a hydrocarbon environment.

Figure 2. Post Mortem Y267 EPDM Seal After 5 Months in Bakersfield Continuous Steam Injection Well @ 204C (400F). Seal is Virtually As New.

BACA, NM, GEOTHERMAL HYDRAULIC STIMULATION

Union Geothermal/Republic Geothermal - DOE performed a hydraulic fracture experiment at the Baca power plant demonstration site. The packer was set at about 915M (3000 ft.) where the formation is normally about 232C (450F). Two Otis packers with L'Garde Y267 EPDM elements were used for the fracture and then subsequently for drill stem tests.

Normally for stimulations with temperatures above 149-177C (300-350F), Republic Geothermal takes special precautions to protect the marginal seal elements made from standard elastomer compounds⁶. They first cool the well, circulate cold water while the packer is being run, leave the packer unset until the moment it is needed so that cold water circulation can be continued, and then just prior to pumping the packer is set.

The above procedure required for packers with standard elastomer compounds is unnecessary for geothermal packers with Y267 EPDM elements deployed in wells even above 260C (500F). Keeping the packer in the balancing act through this crucial period just prior to pumping is eliminated since the packer can be set when it is run, checked out for leakage, and then forgotten, thus freeing personnel to concentrate on other critical problems. In addition, if the standard elastomeric packers fail to pack-off just before pumping, expensive equipment must stand by until successful sealing is accomplished. Hypothetically, for the Baca case, if a standard elastomeric packer would have been run and if it had to be replaced, it would have cost about \$18,000 in standby time.

The Baca well was cooled somewhat when the stimulation packer was run since cold water was circulated to assure that the well would remain killed. Hence, the maximum temperature the stimulation packer experienced was 160C (320F) during the 12 hours it was packed off. The drill stem test packer only saw 171C (340F) because of the cooling from 8000 barrels of fracture fluids and it remained packed off for 18.5 hours.

As with the Bakersfield packers, both were retrieved on the first attempt with no problems whatsoever, textbook retrievals. Both packer seals looked as new which was expected at these temperatures for the Y267 EPDM since this is well below its capability. Figure 3 shows segments of the hydraulic fracture and drill stem test seals next to a new seal. Figure 4 shows the hydraulic fracture packer just after removal from the well. However, these same conditions are considered to be a challenge for standard elastomer compounds and Republic commented that even with precautions taken with standard elastomers under these same conditions, they would expect the post mortem seals to be extruded and/or cracked.

LOS ALAMOS NATIONAL LABORATORY (LANL) CABLEHEAD TESTS⁷

LANL is the prime contractor on a major DOE geothermal demonstration, the Hot Dry Rock Project. They have shown feasibility of the concept at 200C (392F) and are currently preparing to demonstrate the concept with deeper wells and higher

Figure 3. Post Mortem Y267 EPDM Seals After 12 Hrs. in Baca Geothermal Hydraulic Fracture @ 160C (320F) and 18.5 Hrs. in Drill Stem Tests @ 171C (340F). Seals Are As New and Are Compared to a New Seal Element.

Figure 4. Post Mortem Packer With Y267 EPDM Seal After Baca Hydraulic Fracture. Seal Is As New.

temperatures, 275C (525F) minimum. Because of the higher temperatures LANL is concerned about elastomeric parts in various well and logging tools. They awarded L'Garde a contract to support them with the developed high-temperature elastomer expertise.

L'Garde fabricated O-rings and a cable bend protector from the Y267 EPDM packer seal compound for the LANL cablehead. LANL tested cablehead O-rings in an autoclave with water and Mobil One oil. They ran 24-hr. cycles with the temperature and pressure on for 8 hours and off for 16, this simulates tripping in and out of the hole. The best prior performance they achieved was with commercial fluoroelastomer O-rings which literally disintegrated after one cycle.

LANL ran the Y267 EPDM in water for 5 cycles or 5 days before stopping the test to examine the seals. This included a 24-hr. run on the fifth day which provided a total test time of about 56 hours for this one seal. The temperature was nominally 275C (525F) and the pressure was nominally 51.7 MPa (7500 psi). The O-ring looked excellent after this test and obviously could have continued further cycling.

They also ran the Y267 EPDM in Mobil One oil for 4 cycles or 4 days and stopped the tests to examine the seals. The temperature was nominally 275C (525F) with an excursion to 380C (716F) during

one of the 24-hr. cycles. LANL estimates that the seal was exposed to approximately 340C (644F). The pressure was nominally 51.7 MPa (7500 psi). The seals looked good and were sealing after 4 cycles, although there was some swelling from the high-temperature oil and some permanent deformation as would be expected for these conditions. They hope to go ten cycles operationally before they must change out the O-rings.

These tests are landmark tests because this is the first time the Y267 EPDM was tested to the 51.7 MPa (7500 psi) differential pressure range, the first time the Y267 EPDM was tested in 100% oil, the first time the Y267 EPDM was tested in oil at extreme temperatures (excursions to 340C (644F)), and the first time the Y267 EPDM was subjected to a cycling environment. The above results are even further underlined when considering the fact that EPDM's are generally not used in oil environments because they are extremely vulnerable to degradation and swelling. These tests at the extremely high temperatures provide substantiating evidence that the Y267 EPDM is extremely serviceable in oil.

FENTON HILL, NM, LOGGING IN EE-2

Subsequent to the autoclave testing described in the previous section, LANL equipped their high temperature cablehead with L'Garde Y267 EPDM O-rings and cable bend protector. The cablehead was run with a temperature probe into the new EE-2 well which is about 4600 M (15,000 ft.) deep with a bottomhole temperature of 317C (603F). The longest service experienced by Y267 EPDM parts at this writing is bottomhole for 10 hours and above 200C (400F) for 15 hours. The parts sealed and looked excellent after this set of round trips with the most major problem being only some permanent deformation of the O-rings. LANL plans to try for as many as 10 trips on a single set of elastomeric parts when the opportunity avails itself. Figure 5 shows the O-rings which were downhole in the 317C (603F) EE-2 well.

Figure 5. Post Mortem Y267 EPDM Cablehead O-Rings After Multiple Trips Into 317C (603F) EE-2 Well.

TECHNOLOGY TRANSFER

Toward the final stages of the geothermal compound development, it was becoming evident that the technology developed represented significant progress in high-temperature elastomer capability. Usually efforts such as this are concluded with publication and distribution of a final report. However, in this instance the DOE felt it worthwhile to expend additional effort to transfer the technology to industry to further assure that benefit to the industry will be more generally widespread.

Applications for technology transfer were solicited from over 300 companies. There were advertisements placed in the Geothermal Energy Magazine and the Petroleum Engineer International, and from the applications three companies were selected for technology transfer and three additional companies were placed on a back-up list should funds become available. The selected companies are: BJ-Hughes Rubber, Oncor Drilling Tools (Precision Rubber), and Parker Seal. The back-up companies are: NL Rig Equipment, Tri-State Oil Tool, and Vernay Laboratories. In that Oncor Drilling Tools purchases their molded rubber parts from vendors, they have selected Precision Rubber to receive the technology in their behalf.

371C (700F) PACKER DEVELOPMENT

Throughout the development of the casing packer elastomer compounds¹, L'Garde had the opportunity to observe failure of over 100 packer seals. During this period L'Garde developed a design approach which would yield higher temperature and pressure capability for packer systems through minimizing the stresses the seal element experiences.

An opportunity to apply this design approach to hardware and a specific application emerged about 2 years ago. L'Garde invented the MIN-STRESS II casing packer concept which has been under development since that time. The packer requirements include:

Temperature 371C (700F)
Pressure 20.7 MPa (3000 psi)
Duty Cycle, Minimum 1 year

Full scale prototype development of the MIN-STRESS II packer is currently underway.

CONCLUSION

Recent downhole laboratory trials of L'Garde's Y267 EPDM were successful. Trials include the applications which are shown in Table I. Based on

Application	Fluid	Temperature	Pressure	Time
Otis Lab Packer Tests	H ₂ O	302C(575F)	20.7MPa(3000 psi)	5 days
Bakersfield Geot. Steam Injection	Steam/Brine	204C(400F)	1.7MPa(250 psi)	5 mos.
Race Hydraulic Simulation	H ₂ O	160C(320F)	21.0MPa(3000 psi)	32 hours
Race Drill Stem Tests	H ₂ O	171C(340F)	6.3MPa(900 psi)	18.5 hours
LAB. Autoclave Tests	H ₂ O/Brine-1 One	275C(525F)	51.7MPa(7500 psi)	6-8 cycles
LAB. Autoclave, EC-2 Well	H ₂ O	317C(603F)	51.0MPa(7500 psi)	15 hours

Table I. Summary of Downhole and Related Lab Tests

these experiences, the Y267 EPDM provides potential for immediate improvements in the state-of-the-art of geothermal downhole well tool elastomers. In addition to the elastomeric materials improvements, L'Garde is currently developing a new casing packer concept which should provide reliable long-term high temperature casing packer capability to 371C (700F).

ACKNOWLEDGEMENTS

The effort represented by this paper was sponsored by the United States Department of Energy, Division of Geothermal Energy. Mr. J. V. Walker and Dr. R. R. Reeber were instrumental in providing the opportunity to engage in this research and development, and provided valuable guidance throughout the effort. In addition, Dr. P. E. Cassidy of Texas Research Institute contributed significantly as technical coordinator for DOE as did Mr. A. J. Adduci of the San Francisco office of DOE as the technical administrator. Dr. L. E. Kukacka is currently providing valuable guidance on the Technology Transfer effort.

Invaluable information was obtained through the cooperation of several Government laboratories and commercial companies in the oil and gas, and chemical products industries. Mr. J. K. Sieron of the Air Force Materials Laboratory and Dr. J. C. Rowley of Los Alamos National Laboratory were especially helpful.

REFERENCES

- Hirasuna, A.R., et al., July 1979, "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976 - 30 June 1979," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy, Geothermal Energy.
- Hirasuna, A.R., et al., "Important Conclusions Based on Results of Geothermal Casing Packer Seal Elastomers Testing," presented at a meeting of the Geothermal Resources Council, Hilo, Hawaii, 25-27 July 1978, Summary in Transactions, page 279.
- Hirasuna, A.R., et al., "Results of Geothermal Casing Packer Seal Elastomer Compound Development," presented at a meeting of the Geothermal Resources Council, Reno, Nevada, September, 1979, Summary in Transactions, page 309.
- Friese, G.J., "Elastomer Compatibility With Brine, Isobutane, and Oil," submitted for presentation at the 1981 Annual Meeting, Geothermal Resources Council, 25-29 October 1981, Houston, TX.
- Taylor, D., Private Communication, Otis Engineering Corp., Dallas, Texas.
- Verity, R., Private Communication, Republic Geothermal, Santa Fe Springs, CA.
- Archuleta, J. and Dennis, B., Private Communication, Los Alamos National Laboratory, Los Alamos, NM.

HIGH TEMPERATURE Y267 EPDM ELASTOMER
FIELD AND LABORATORY EXPERIENCES, AUGUST 1981

Alan R. Hirasuna
Gilbert J. Friese
Clifford A. Stephens

L'Garde, Inc.
Newport Beach, California 92663

INTRODUCTION

In 1976, reliable elastomers for the unusually severe geothermal environment at 260C (500F) did not exist. L'Garde, Inc. developed geothermal elastomer compounds during the period of 1976-1979 under U. S. Department of Energy-Division of Geothermal Energy (DOE-DGE) contract DE-AC03-77ET28309 [1]. The resulting developments yielded compounds from four polymer systems which successfully exceeded the contract requirements.

Since completion of the compound development, significant laboratory and downhole experience occurred, primarily on the Y267 EPDM compound. This paper summarizes those experiences which firmly indicate a superior elastomer for high-temperature brines and other environments.

ELASTOMER COMPOUND DEVELOPMENT 1976-1979

Previous elastomer compound development by L'Garde resulted in compounds from four different polymer systems which exceeded the original contract requirements. The polymer systems include: EPDM (Nordel), FKM (Viton), EPDM/FKM Blends, and propylene/TFE (AFLAS). The packer requirements include the following environments:

260C (500F)
20.7-27.6 MPa (3000-4000 psi)
Sour brine
24 hours

Though compounds from all four polymer systems exceeded the requirements, one of the compounds is particularly successful because of its good processing qualities, extremely good thermochemical stability, extremely good mechanical properties, and finally its low cost constituents. The compound, Y267 EPDM, now has significant laboratory and downhole experience which are the case histories given below. An additional virtue of the compound is its uncharacteristically good performance (for EPDM's) in hydrocarbons. Three experiences are quoted

below, where Y267 EPDM was exposed to oil at high temperatures.

OTIS ENGINEERING CORP. PACKER TESTS

Y267 EPDM packer seals were molded by L'Garde for tests by Otis Engineering Corp. [2]. The seals are for a 7-inch casing packer which Otis tested in 302C (575F) and 20.7 MPa (3000 psi) differential pressure water in their laboratory simulation tester. The elements were successfully sealing after 5 days when the test was terminated. The seal was in excellent condition considering the environment and did not show any signs of reversion softening or scission. Figure 1 shows the post mortem seal.

Prior to this test Otis tested an EPDM from another firm but at a lower temperature, 288C (550F), and the seal failed because it lost strength due to reversion softening of the polymer. The post mortem seal did not have any form and looked like a glob of tar. Otis has since tried several other competing EPDM compounds and none has yet equaled the performance of the Y267 EPDM.

BAKERSFIELD CONTINUOUS STEAM INJECTION

The Y267 EPDM element on an R&D Otis packer was fielded in a Bakersfield heavy oil continuous steam injection well. The steam is 246C (475F) at the boiler and 204C (400F) at the wellhead.

The packer was pulled after 5 months of continuous steaming because the well required sand removal. Tracer tests confirmed that the packer was still sealing prior to removal. Typically, difficulty is encountered in retrieving thermal packers but this packer was retrieved with no problems whatsoever, where usually difficulty is encountered with the seal bonding to the casing, or hardening in place requiring the seal to be broken to enable packer retrieval.

The appearance of the recovered

seal was excellent. It looked virtually as new after 5 months continuous operation and it was apparent that it could have remained in operation indefinitely. Figure 2 is a photograph of the packer seal after it was removed from the Bakersfield well. The good condition is particularly significant since the Y267 EPDM seal was in contact with crude oil which covered the packer and element when it was retrieved. EPDM's are generally considered inappropriate for a hydrocarbon environment.

BACA, NEW MEXICO GEOTHERMAL HYDRAULIC STIMULATION

Union Geothermal/Republic Geothermal - DOE performed a hydraulic fracture experiment at the Baca power plant demonstration site. The packer was set at about 915M (3000 ft.) where the formation is normally about 232C (450F). Two Otis packers with L'Garde Y267 EPDM elements were used for the fracture and then subsequently for drill stem tests.

Normally for stimulations with temperatures above 149-177C (300-350F), Republic Geothermal takes special precautions to protect the marginal seal elements made from standard elastomer compounds [3]. They first cool the well, circulate cold water while the packer is being run, leave the packer unset until the moment it is needed so that cold water circulation can be continued, and then just prior to pumping the packer is set.

The above procedure required for packers with standard elastomer compounds is unnecessary for geothermal packers with Y267 EPDM elements deployed in wells even above 260C (500F). Keeping the packer in the balancing act through this crucial period just prior to pumping is eliminated since the packer can be set when it is run, checked out for leakage, and then forgotten, thus freeing personnel to concentrate on other critical problems. In addition, if the standard elastomeric packers fail to pack-off just before pumping, expensive equipment must stand by until successful sealing is accomplished. Hypothetically, for the Baca case, if a standard elastomeric packer would have been run and if it had to be replaced, it would have cost about \$18,000 in standby time.

The Baca well was cooled somewhat when the stimulation packer was run since cold water was circulated to assure that the well would remain killed. Hence, the maximum temperature the stimulation packer experi-

enced was 160C (320F) during the 12 hours it was packed off. The maximum pressure during stimulation was 21.2 MPa (3080 psi). The drill stem test packer only saw 171C (340F) and 4.1 MPa (600 psi) because of the cooling from 8000 barrels of fracture fluid and it remained packed off for 18.5 hours.

As with the Bakersfield packers, both were retrieved on the first attempt with no problems whatsoever, textbook retrievals. Both packer seals looked as new which was expected at these temperatures for the Y267 EPDM since this is well below its capability. Figure 3 shows segments of the hydraulic fracture and drill stem test seals next to a new seal. Figure 4 shows the hydraulic fracture packer just after removal from the well. However, these same conditions are considered to be a challenge for standard elastomer compounds and Republic commented that even with precautions taken with standard elastomers under these same conditions, they would expect the post mortem seals to be extruded and/or cracked.

LOS ALAMOS NATIONAL LABORATORY (LANL) CABLEHEAD TESTS [4]

LANL is the prime contractor on a major DOE geothermal demonstration, the Hot Dry Rock Project. They have shown feasibility of the concept at 200C (392F) and are currently preparing to demonstrate the concept with deeper wells and higher temperatures, 275C (525F) minimum. Because of the higher temperatures LANL is concerned about elastomeric parts in various well and logging tools. They awarded L'Garde a contract to support them with the developed high-temperature elastomer expertise.

L'Garde fabricated O-rings and a cable bend protector from the Y267 EPDM packer seal compound for the LANL cablehead. LANL tested cablehead O-rings in an autoclave with water and Mobil One oil. They ran 24-hour cycles with the temperature and pressure on for 8 hours and off for 16, this simulates tripping in and out of the hole. The best prior performance they achieved was with commercial fluoroelastomer O-rings which literally disintegrated after one cycle.

LANL ran the Y267 EPDM in water for 5 cycles or 5 days before stopping the test to examine the seals. This included a 24-hour run on the fifth day which provided a total test time of about 56 hours for this one seal. The temperature was nominally 275C (525F) and the pressure was nominally 51.7 MPa (7500 psi). The O-ring

looked excellent after this test and obviously could have continued further cycling.

They also ran the Y267 EPDM in Mobil One oil for 4 cycles or 4 days and stopped the tests to examine the seals. The temperature was nominally 275C (525F) with an excursion to 380C (716F) during one of the 24-hour cycles. LANL estimates that the seal was exposed to approximately 340C (644F). The pressure was nominally 51.7 MPa (7500 psi). The seals looked good and were sealing after 4 cycles, although there was some swelling from the high-temperature oil and some permanent deformation as would be expected for these conditions. They hope to go ten cycles operationally before they must change out the O-rings.

These tests are landmark tests because this is the first time the Y267 EPDM was tested to the 51.7 MPa (7500 psi) differential pressure range, the first time the Y267 EPDM was tested in 100% oil, the first time the Y267 EPDM was tested in oil at extreme temperatures (excursions to 340C (644F)), and the first time the Y267 EPDM was subjected to a cycling environment. The above results are even further underlined when considering the fact that EPDM's are generally not used in oil environments because they are extremely vulnerable to degradation and swelling. These tests at the extremely high temperatures provide substantiating evidence that the Y267 EPDM is extremely serviceable in oil.

FENTON HILL, NEW MEXICO
LOGGING IN EE-2

Subsequent to the autoclave testing described in the previous section, LANL equipped their high temperature cablehead with L'Garde Y267 EPDM O-rings and cable bend protector. The cablehead was run with a temperature probe into the new EE-2 well which is about 4600M (15,000 ft.) deep with a bottomhole temperature of 317C (603F). The longest service experienced by Y267 EPDM parts at this writing is bottomhole for 10 hours and above 200C (400F) for 15 hours. The parts sealed and looked excellent after this set of round trips with the most major problem being only some permanent deformation of the O-rings. LANL plans to try for as many as 10 trips on a single set of elastomeric parts when the opportunity avails itself. Figure 5 shows the O-rings which were downhole in the 317C (603F) EE-2 well.

BINARY PLANT COMPATIBILITY TESTS [5]

Because of the absence of compatibility data of high performance elastomers, significant expense is often incurred as each individual project attempts to find the right elastomer for its requirements by trial and error. Recognizing this the DOE/Brookhaven National Laboratory contracted L'Garde to run compatibility tests for elastomers for the binary power plant application.

A typical plant might be located in the Imperial Valley in an area where the brine temperature is 191C (375F) and use isobutane/isopentane as the working fluids. In addition, there may be other equipment such as downhole electrical pumps which need a high temperature oil. Hence, static seal compatibility tests were run for synthetic brine, isobutane (represents isopentane also), and oil at nominally 191C (375F) with some testing up to 266C (510F). The oils tested are ASTM No. 1, ASTM No. 3, Chevron Cylinder 460X, and Pacer DHT-185M.

Given the test conditions, it was doubtful that any one compound would work satisfactorily in all three fluids. Nevertheless, this was highly desirable and the testing was structured to reveal this should any compound have that capability. Several of the molded rubber product manufacturers were solicited for their most promising compound(s) for the test. Ultimately 34 compounds from 15 different companies were selected for testing. A full spectrum of high temperature polymers were included: EPDM, Kalrez, PNF, Viton/Fluorel, Nitrile/Buna N, and AFLAS.

Immersion testing in each of the three fluids were run to screen the 34 candidates down to 8 for further evaluation. The immersion tests were run at 191C (375F) for 5 days and evaluated on the basis of change in hardness, ultimate tensile strength, ultimate elongation, weight, volume, and resilience. This screening down process was done while keeping aware that immersion tests only test the effect of temperature and chemistry on the elastomer. At these higher temperatures, the mechanical environment due to differential pressures, seal setting, etc., are just as important, if not moreso, than temperature and chemistry. Not surprising, what looked the best based on immersion tests did not look the best after full-environment static seal tests.

After selection, the 8 compounds were then further evaluated. They were immersion tested at 191C (375F) for longer periods, and tested as O-rings up to 266C (510F). Based on these tests 4 compounds will be O-ring tested at 204C (400F) and 20.7 MPa (3000 psi) for 6 months. The overall summary matrix of tests is shown in Table I.

Figure 6 summarizes the short-term O-ring tests which is a representative summary of all of the testing done to date. Only 6-month O-ring tests are yet to be done. All testing was done at 20.7 MPa (3000 psi) and Figure 6 shows only the EPDM's performing reliably up to the 266C (510F) level. Catastrophic failure occurred with many of the compounds especially in isobutane and brine. The AFLAS 7170X14 and Kalrez 1018 both catastrophically failed at the minimum test temperature, 191C (375F), in isobutane. The E692-75 EPDM failed in isobutane, hence, does not show across the board reliability that Y267 EPDM shows. Figures 7, 8, and 9 show the Y267 EPDM after testing in the static O-ring test with a differential pressure and at the indicated temperatures. Figures 7, 8, and 9 are for brine, isobutane and oil, respectively. Figure 10 shows the Parker E692-75 EPDM for isobutane. This compound performed quite well in general, however, results indicate that its capability is being approached for the more severe conditions. Similar static O-ring tests in 260C (500F) brine reported in Reference 1, indicate nibbling of the E692-75 in brine at 28.3 MPa (4100 psi). Hence, these conditions are about the upper limit for the E692-75 while the capability of the Y267 EPDM is indicated to be somewhat higher since it has not experienced breakage or nibbling.

In conclusion for static seals, the Y267 EPDM performs the best at 266C (510F). This conclusion is especially unexpected for the hydrocarbon oil environment because it is a generally accepted rule that EPDM's should not be used with hydrocarbon oil. This rule is derived from immersion tests and even though the Y267 EPDM is less vulnerable to oil than most EPDM's, it does swell, and lose strength and hardness. Nevertheless, after several days in the environment the degradation reaches a plateau and the remaining properties still provide an excellent static seal and, in fact, the best for binary plant environments.

OTHER MISCELLANEOUS TESTS

Other miscellaneous tests on Y267 EPDM have been run for a variety of environments and by several different organizations. These are summarized in this section.

Battelle Northwest [6] ran into rubber seal problems on an inline corrosion probe they are testing. They ran autoclave tests in 200C (400F) brine on the Y267 EPDM prior to installing in the field. They have tested several compounds and confirm that the Y267 EPDM stands up better than any other elastomer they have tested. They subsequently ran excellent comparative tests at the Magma Plant in the Imperial Valley. A test loop is available at the feed to the plant where several instruments can be tested simultaneously. Battelle ran corrosion probes with fluoroelastomer (FKM), per fluoroelastomer (FFKM), and Y267 EPDM seals side-by-side. Both the FKM and FFKM seals failed within 2 weeks, the FKM became brittle, and the FFKM cracked and split. The Y267 EPDM is still running at this writing, over 6 months. This is the longest performance logged for this application for any elastomeric seal.

LANL/Union Oil [7] are running a blast stimulation experiment at the Geysers. They have encountered seal problems with their vessel which is immersed in the proprietary explosive. Immersion tests were subsequently run in the explosive at 246C (475F) and 6.9 MPa (1000 psi) with four different elastomer compounds and the Y267 EPDM was clearly the best performer. Tests were also successfully run in 260C (500F) water in an autoclave. These Y267 EPDM seals are now scheduled to go downhole at the Geysers.

LANL [8] ran some temperature surveys for Union at the Baca using their cablehead equipped with Y267 EPDM seals. The well temperature was approximately 300C (575F) and the Y267 EPDM parts were exposed for a maximum of 4 to 6 hours. The rubber parts were changed whenever the cablehead was dismantled for other reasons.

Sandia [9] ran autoclave tests in the vapor phase of water at 270C (500F) for 100 hours plus heat-up and cool-down. The cable BOP environment was being simulated. The Y267 EPDM was the best of 7 compounds in this environment. It retained about 90% of its tensile strength after ageing and was recommended by Sandia for this application.

Spery Vickers [10] ran long-term compatibility tests of elastomers in an aqueous solution of hydrazine at 218C (425F). They saw no change in the Y267 EPDM after 85 days.

TerraTek [11] ran drill bit seal tests on several elastomers including Y267 EPDM. The test exposes the seal to grease on one side and rock cuttings, sand, and water on the other. The specimen is raised to a presoak temperature to simulate tripping into the well, cooled to 150C (302F) to simulate cold drilling fluid, and then the dynamic test is initiated. The test provides a rotating motion and on each cycle a jog is induced in the rotation. At the time they ran Y267 EPDM, a 40 to 50 hour run was quite respectable. With the Y267 a presoak temperature of 288C (550F) was chosen, this is the first time at this high a test temperature. Both TerraTek and L'Garde were pleasantly surprised when the Y267 EPDM was still sealing after 104 hours. TerraTek remarked that the Y267 appeared excellent after the test, less degraded than the other elastomers tested.

AVAILABILITY OF Y267 EPDM

A major objective of the DOE effort was to assure that the developed technology is available for widespread field use. To this end, L'Garde was contracted to transfer the technology. After an application and evaluation process BJ-Hughes Rubber, Oncor/Precision Rubber, and Parker Seal were selected for transfer. The transfer is now in the final stages and molded parts based on Y267 EPDM technology are becoming available in commercial quantities from BJ-Hughes Rubber, Parker Seal, and Precision Rubber. The full spectrum of large and small parts are available from these suppliers. In addition, the technology is available on an R&D basis directly from L'Garde.

CONCLUSION

Downhole and laboratory trials of L'Garde's Y267 EPDM were successful. Table II summarizes those 14 experiences and they indicate that Y267 EPDM is the superior elastomer for high temperature aqueous environments. Test data is also building which indicates superiority for high temperature hydrocarbon oil environments for static seals, isobutane and hydrazine environments.

Parts based on Y267 EPDM technology are available in commercial quantities from

BJ-Hughes Rubber
Parker Seal
Precision Rubber

Y267 EPDM technology is available on an R&D basis directly from L'Garde.

ACKNOWLEDGEMENTS

The effort represented by this paper was sponsored by the United States Department of Energy, Division of Geothermal Energy. Mr. J. V. Walker and Dr. R. R. Reeber were instrumental in providing the opportunity to engage in this research and development, and provided valuable guidance throughout the effort. In addition, Dr. P. E. Cassidy of Texas Research Institute contributed significantly as technical coordinator for DOE as did Mr. A. J. Adduci of the San Francisco office of DOE as the technical administrator. Dr. L. E. Kukacka of Brookhaven National Laboratory is currently providing valuable guidance on the Technology Transfer to industry and the Binary Plant Compatibility Testing Program.

Invaluable information was obtained through the cooperation of several Government laboratories and commercial companies in the oil and gas, and chemical products industries. Mr. J. K. Sieron of the Air Force Materials Laboratory and Dr. J. C. Rowley of Los Alamos National Laboratory were especially helpful.

REFERENCES

- [1] A. R. Hirasuna, et al., July 1979, "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976 - 30 June 1979," L'Garde, Inc., Newport Beach, CA 92663, SAN/1308-2, U.S. Department of Energy, Geothermal Energy.
- [2] D. Taylor, Private Communication, Otis Engineering Corp., Dallas, TX.
- [3] R. Verity, Private Communication, Republic Geothermal, Santa Fe Springs, CA.
- [4] J. Archuleta and B. Dennis, Private Communication, Los Alamos National Laboratory, Los Alamos, NM.
- [5] G. J. Friese, "Elastomer Compatibility With Brine, Isobutane, and Oil", 1981 Annual Meeting, Geothermal Resources Council, 25-29 October 1981, Houston, TX.
- [6] D. Shannon, M. Danielson, Private Communication, Battelle Northwest, Richland, WN.

[7] C. Allen, Private Communication, Union Oil Research, Brea, CA.

[10] D. Nowicki, Private Communication, Sperry Vickers, Jackson, MS.

[8] B. Dennis, B. Todd, Private Communication, Los Alamos National Laboratory, Los Alamos, NM.

[11] R. Hendrickson, R. Winzenreid, Private Communication, TerraTek, Salt Lake City.

[9] C. Arnold, Jr., 19 August 1979, Memo to Distribution, Sandia National Laboratories, Albuquerque, NM.

Table I. Summary Matrix, Binary Power Plant Compatibility Testing

Number Tested	Immersion (6 Fluids)			O-ring (3 Fluids)	
	5d	2 mos.	6 mos.	2d	6 mos.
34 Compounds 191C (375F)	X				
8 Selected 191C (375F) 232C (450F) 266C (510F)		X	X	X X X	
4 Selected (Future) 204C (400F)					X

Table II. Summary of Y267 EPDM Laboratory and Field Experience

Test	Fluid	Temperature C (F)	Pressure MPa (PSI)	Time		
				Hrs.	Days	Mos.
Otis Lab, Packer	H ₂ O	302 (575)	20.7 (3000)		5	
Bakersfield, Packer	H ₂ O, Crude	204 (400)	1.7 (250)		150	5
Baca Stim, Packer	Frac.	160 (320)	21.2 (3080)	12	0.5	
Baca DST, Packer	Frac/H ₂ O	171 (340)	4.1 (600)	18.5	.8	
LANL Lab, O-ring	H ₂ O/Oil	275 (525)	51.7 (7500)	40	1.7	
Fenton Hill, O-ring	H ₂ O	317 (603)	41.4 (6000)	10	0.4	
L'Garde Lab, O-ring	Brine/Oil/Butane	266 (510)	20.7 (3000)	48	2	
Battelle Lab	Brine	200 (392)	4.1 (600)		4	
Magma, Seal	Brine	180 (356)	N/A (N/A)		180+	6+
Union Lab	Explosive	246 (475)	6.9 (1000)	24	1	
Baca, Log O-ring	H ₂ O	3000 (575)	11.7 (1700)	6	.3	
Sandia Lab	H ₂ O	260 (500)	4.7 (680)	100	4.2	
Sperry Lab	H ₂ O, Hydrazine	218 (425)	2.2 (325)	85	3.5	
TerraTek Lab, O-ring	H ₂ O, Debris/Grease	288 (550)	1.4 (0/200)	104	4.3

Figure 1. Post Mortem Y267 EPDM Seal After 5 Days of Testing @ 302C (575F) and 20.7 MPa (3000 psi). Note the Excellent Condition Considering the Extreme Severity of the Environment.

Figure 2. Post Mortem Y267 EPDM Seal After 5 Months in Bakersfield Continuous Steam Injection Well @ 204C (400F). Seal is Virtually as New.

Figure 3. Post Mortem Y267 EPDM Seals After 12 Hours in Baca Geothermal Hydraulic Fracture @ 160C (320F) and 18.5 Hours in Drill Stem Tests @ 171C (340F). Seals are as New and Are Compared to a New Seal Element.

Figure 5. Post Mortem Y267 EPDM Cablehead O-Rings After Multiple Trips Into 317C (603F) EE-2 Well at 4600M (15,000 Ft.).

Figure 4. Post Mortem Packer With Y267 EPDM Seal After Baca Hydraulic Fracture. Seal is as New.

Figure 6. O-Ring Test Results

20.7 MPa (3000 PSI) ΔP

Compound	Rank	Temperature		
		191C(375F)	232C(450F)	266C(510F)
L'Garde EPDM Y267	1		Brine
	1		Isobutane
	1		ASTM #1 Oil
Parker EPDM E692-75	1
	2
	2
L'Garde Viton 501	4		Brine
	5		Isobutane
	3		ASTM #1 Oil
Vernay Lab Fluorel 16959	7
	4
	3
Precision Fluorel VL1503M3	8		Brine
	3		Isobutane
	5		ASTM #1 Oil
Seals Eastern AFLAS 7170X14	3
	7
	6
L'Garde AFLAS 291	4		Brine
	5		Isobutane
	8		ASTM #1 Oil
DuPont Kalrez 1018	4
	7
	7

Damage limited to moderate set and swelling.

Severe set, cracks or splits, signs of extrusion.

Nibbling or catastrophic failure.

Figure 7. L'Garde Y267 EPDM After 2 Days in Brine at 20.7 MPa (3000 psi).

Figure 8. L'Garde Y267 EPDM After 2 Days in Isobutane at 20.7 MPa (3000 psi).

Figure 9. L'Garde Y267 EPDM After 2 Days in ASTM No. 1 Oil at 20.7 MPa (3000 psi).

Figure 10. Parker E692-75 EPDM After 2 Days in Isobutane at 20.7 MPa (3000 psi).

IADC/SPE 11407

A Proven Elastomer Compound for Extremely Hostile Geothermal and Oilfield Environmentsby A.R. Hirasuna, G.J. Friese, and C.A. Stephens, *L'Garde, Inc.*

This paper was presented at the IADC/SPE 1983 Drilling Conference held in New Orleans, Louisiana, February 20-23, 1983. The material is subject to correction by the author. Permission to copy is restricted to an abstract of not more than 300 words. Write SPE, 6200 North Central Expressway, Drawer 64706, Dallas, Texas 75206.

ABSTRACT

Since 1979 the Y267 EPDM elastomer has been independently tested by other organizations in a variety of field and laboratory applications. The following are some examples. The same Y267 EPDM O-rings worked with no leaks as logging tool seals for multiple trips to 4600M (15k ft.) at 320C (608F) BHST. A packer element performed flawlessly for five months in a 204C (400F) continuous steam injection well and was retrieved at the end of the test in an as-new condition. A high-pressure Y267 EPDM packer test was performed with complete success at 232C (450F) for a 7.5 day test in sour crude with differential pressures to 138 MPa (20 ksi) and the seal condition was only very slightly changed by the test. Comprehensive compatibility testing of 34 compounds from 15 companies in geothermal brine, isobutane, and oil at 191C-266C (375-510F) showed that the Y267 EPDM was best of the 34 in all three fluids.

Over 15 laboratory and over 20 field case histories of Y267 EPDM such as the above examples are reported. All strongly establish that Y267 EPDM is at the cutting edge of technology.

INTRODUCTION

During the period of 1976 to 1979 under the sponsorship of the U.S. Department of Energy - Division of Geothermal Energy, L'Garde, Inc. developed new elastomer compounds for the unusually hostile 260C (500F) geothermal environments. Though compounds based on four different polymer systems exceeded the contract requirements, one compound (Y267 EPDM) was eminently successful for high-temperature geothermal, and oil and gas applications.

Since completion of the compound development, widespread laboratory and downhole experience occurred. This paper summarizes over 15 laboratory and over 20 field tests of the Y267 EPDM under various extreme conditions. 95% of the tests were performed independent of L'Garde by other organizations. These and other substantiating data clearly show that

References and illustrations at end of paper.

Y267 EPDM can be classified as a technology breakthrough for high-temperature elastomers.

EPDM IS NOT EPDM

Unfortunately, it has been common in the industry to specify an elastomer by the polymer and its hardness, e.g. 70 durometer nitrile. This is adequate for benign environments, however, in hostile environments which are becoming more and more commonplace today, this simple specification is grossly inadequate.

Generally speaking an elastomer compound is made up of about 50% polymer, i.e. EPDM, and about 50% other ingredients. Hence, there is wide latitude to vary the characteristics with the other 50%, and it is not unusual for one of the very minor ingredients to markedly change the characteristics. In addition, with EPDMs there are about seven American manufacturers and each has about ten different EPDM polymers which have varying characteristics. It is true that a polymer will afford general characteristics virtually independent of the specific compound, however, when the requirements demand the ultimate capability to be yielded, the specific compound is critical and very probably specific tailoring for the application is required. Fortunately, the Y267 EPDM is showing superior performance in a wide variety of applications beyond the original intent, static geothermal seals. When Y267 EPDM works well outside the realm for which it was designed, the opportunity remains for even better performance with specific tailoring as was done for the geothermal static seals. Given this insight, it becomes obvious that specifying polymer and hardness is grossly inadequate for hostile environments, i.e. an EPDM is not an EPDM.

Y267 EPDM specifies a recipe and a manufacturing procedure, both of which are critical to its ultimate performance. There have been several direct comparisons between Y267 EPDM technology and other EPDM compounds by L'Garde and other companies all indicating the superiority of Y267 EPDM. Otis Engineering Corp. tested over ten

other EPDM compounds from three companies as 7-inch packer seals, none of which compared favorably to Y267 EPDM. Parker Seals directly compared their previous generation E692-75 EPDM against their new E962-85 EPDM which is based directly on the Y267 EPDM technology. Table I shows the characteristics after ageing in steam at 288C (550F) and 316C (600F) for 168 hours. Significant differences show up at 288C and complete deterioration of the old EPDM occurs at 316C.

SUMMARY OF CASE HISTORIES

This paper is dedicated to a comprehensive summary of the most pertinent experiences with Y267 EPDM. Details such as the development and photographs showing as-new condition of post-mortem parts are not included in favor of a more comprehensive summary of case histories within the allotted space. Omitted details can be obtained in References 3 through 5.

Table II summarizes the most important known experiences with Y267 EPDM. Those cases are divided into laboratory and field tests. Over 15 laboratory tests and over 20 field trials are listed. The related laboratory tests which were preliminary to field trials are grouped side-by-side along with the critical conditions. Note that there are temperatures in excess of 316C (600F), pressures up to 207 MPa (30,000 psi), times in excess of eight months, and in various fluids including brine, sour crude, antioxidants, oil, etc. Virtually all parts tested were made by L'Garde, Inc., BJ-Hughes Rubber Division, Parker Seals, or Precision Rubber. Except for three laboratory tests, all others were performed by organizations independent of the fabricators.

The balance of the paper is devoted to details on specific case histories.

BINARY PLANT ELASTOMER COMPATIBILITY TESTS (ECT)

Because of the absence of adequate compatibility data of elastomers for hostile applications, significant expense is often incurred as each individual project attempts to find the right elastomer for its requirements by trial and error. Recognizing this the DOE/Brookhaven National Laboratory contracted L'Garde to run compatibility tests for elastomers for the binary geothermal power plant application.

A typical plant might be located in the Imperial Valley, in an area where the brine temperature is 191C (375F), and use isobutane/isopentane as the working fluids. In addition, there may be other equipment such as downhole electrical pumps which need a high temperature oil. Hence, static seal compatibility tests were run in synthetic brine, isobutane (represented isopentane also), and four oils at nominally 191C (375F) with some testing up to 266C (510F). The oils tested are ASTM No. 1, ASTM No. 3, Chevron Cylinder 460X, and Pacer DHT-185M. The synthetic geothermal brine was developed during the elastomer development program and contains the following starting constituency:

H ₂ S	300 ppm
CO ₂	1000

NaCl 25,000

H₂O balance

Given the test conditions, it was doubtful that any one compound would work satisfactorily in all three fluids. Nevertheless, this was highly desirable and the testing was structured to reveal this should any compound have that capability. Several of the molded rubber product manufacturers were solicited for their most promising compound(s) for the test conditions. Ultimately 34 compounds from 15 different companies were selected for testing. A full spectrum of high-performance polymers were included: EPDM, Kalrez®, PNF, Viton®/Fluorel®, Nitrile/Buna N, and AFLAS®.

Immersion testing in each of six fluids were run to screen the 34 candidates down to eight for further evaluation. The immersion tests were run at 191C (375F) for five days and evaluated on the basis of change in hardness, ultimate tensile strength, ultimate elongation, weight, volume, and resilience. This screening down process was done with the keen awareness that immersion only tests the effect of temperature and chemistry on the elastomer. At these higher temperatures, the mechanical environment due to differential pressures, seal setting, etc., are just as important, if not moreso, than temperature and chemistry. Not surprisingly, what looked the best based on immersion tests did not look the best after subsequent full-environment static seal tests.

After selection, the eight compounds were then further evaluated. They were immersion tested at 191C (375F) for longer periods, and tested as O-rings up to 266C (510F). Based on these tests, four compounds were O-ring tested at 204C (400F) and 20.7 MPa (3000 psi) for six months. The overall summary matrix of tests is shown in Table III.

Figure 1 summarizes the short-term O-ring tests. All testing was done at 20.7 MPa (3000 psi) differential pressure and Figure 1 shows only the EPDMs performing reliably up to the 266C (510F) level. Catastrophic failure occurred with many of the compounds especially in isobutane and brine. The AFLAS 7170X14 and Kalrez 1018 both catastrophically failed at the minimum test temperature, 191C (375F), in isobutane. The E692-75 EPDM failed in isobutane, hence, does not show across-the-board reliability that Y267 EPDM shows. This compound performed quite well in general; however, results indicate that its capability is being approached for the more severe conditions. Similar static O-ring tests in 260C (500F) brine indicate nibbling of the E692-75 in brine at 28.3 MPa (4100 psi).³ Hence, these conditions are about the upper limit for the E692-75 while the capability of the Y267 EPDM is indicated to be somewhat higher since it has not experienced breakage or nibbling. Note that at higher temperatures the Y267 EPDM is superior in all three fluids.

Six-month, long-term O-ring tests were run completing the compatibility test program. The tests were run for approximately six months at 204C (400F) and 20.7 MPa (3000 psi) differential pressure. The long-term tests were run on a

different fixture than the short-term tests. The short-term tests were strictly static-seal tests while the long-term tests permitted slow sliding, up to a cycle per day. This design was adopted strictly because of economic considerations. The four best compounds (first four in Figure 1) from the short-term O-ring tests were tested. Catastrophic failure occurred in isobutane and brine, again the most difficult fluids to seal. Because of the relatively mild test conditions, 204C, more choices became available, e.g. Y267, E692-75, and 501 all performed satisfactorily in ASTM #1 oil.

In brine for six months, the Y267 looks the best which is not surprising because it was designed for brine. The Parker E692-75 EPDM test fixture failed after 56 days because a non-test O-ring in the fixture failed; but based on previous data, there is every reason to believe that it would have performed equally at this relatively mild temperature. Both the fluoroelastomers became brittle and failed.

In isobutane for six months, both E692-75 EPDM and 501 Viton are "not OK"; they failed at 90 and 41 days respectively consistent with previous tests. There is significant uncertainty, however, as to Y267 EPDM and VL1503M3 Fluorel. For the conditions VL1503M3 is best because it survived the entire test, 171 days, but suffered moderate permanent set, while the Y267 suffered slight permanent set and it cracked over about 10% of its circumference but stayed intact. The Y267 crack may have originated with a molding defect and, in any event, it could probably be prevented with a backup ring. Consequently in the final analysis, these and other factors would enter into the final selection for isobutane, and more testing is needed.

In ASTM #1 oil for six months, a broader choice exists as all four candidates survived the 209-day test period. The VL1503M3 Fluorel became brittle, suffered severe set, and slight nibbling. The EPDMs suffered some swell and moderate nibbling. The 501 Viton suffered severe set and slight nibbling, however, it did not get brittle as did the VL1503M3 or as it did in Chevron Cylinder Oil. A trade exists here between severe set and potential of getting brittle for the 501 Viton, against the moderate nibbling of the EPDMs which can be corrected with backup rings.

These data are particularly valuable because of the comparison between multiple candidates under controlled broad spectrum of environments. In overall conclusion for temperatures in excess of 232C (450F), the Y267 EPDM is the best indicated choice for all three fluids -- brine, isobutane, and ASTM #1 oil. At more benign temperatures below 204C (400F), more choices exist particularly in ASTM #1 oil.

The above conclusions regarding the serviceability of EPDMs in hydrocarbon oil is unexpected because it is a generally accepted rule that EPDMs should not be used with hydrocarbon oil. This rule is derived from immersion tests and even though the Y267 is less vulnerable to oil than most EPDMs, it does swell, and lose strength and hardness. Nevertheless, after less than five days in the environment the degradation reaches a plateau which has been measured to be maintained at least six

months. The plateau properties still provide an excellent static seal. There is some evidence, in fact, that swell may be a desirable property for seals in hostile environments.

LAB PACKER TESTS

Y267 EPDM packer seals were molded by L'Garde for tests by Otis Engineering Corp. The seals are for a 7-inch casing packer which Otis tested in 302C (575F) and 20.7 MPa (3000 psi) differential pressure water in their laboratory simulation tester. The elements were successfully sealing after five days when the test was terminated. The seal was in excellent condition considering the environment and did not show any signs of reversion softening or scission.

Prior to this test Otis tested an EPDM from another firm but at a lower temperature, 288C (550F), and the seal failed because it lost strength due to reversion softening of the polymer. Otis has since tried several other competing EPDM compounds and none has yet equaled the performance of the Y267 EPDM.

In addition to the above tests, Otis also tested Y267 packer seals twice in sweet and once in sour crude. The more severe sweet crude test was at 232C (450F) and 103 MPa (15,000 psi) for six days. The sour crude test went to even higher pressure. It was run at 232C (450F) for three days at 69 MPa (10,000 psi) and then the pressure was increased for 4.5 days to 138 MPa (20,000 psi). Otis reports that the seals were undamaged and just showed only very slight swell and softening. They did not expect any elastomeric seal to hold up at 138 MPa (20,000 psi). They conclude that the Y267 EPDM is a definite candidate for this extraordinarily extreme environment.

CONTINUOUS STEAM INJECTION PACKER

The Y267 EPDM element on an R&D Otis packer was fielded in a Bakersfield heavy oil continuous steam injection well. The steam is 246C (475F) at the boiler and 204C (400F) at the wellhead.

The packer was pulled after five months of continuous steaming because the well required sand removal. Typically, difficulty is encountered in retrieving thermal packers but this packer was retrieved with no problems whatsoever. Normally difficulty is encountered with the seal bonding to the casing or hardening in place.

The appearance of the recovered seal was excellent. It looked virtually as new after five months continuous operation and it was apparent that it could have remained in operation indefinitely. The good condition is particularly significant since the Y267 EPDM seal was in contact with crude oil which covered the packer and element when it was retrieved. EPDMs are generally considered inappropriate for hydrocarbon environments.

GEOTHERMAL HYDRAULIC STIMULATION PACKER⁸

Union Geothermal/Republic Geothermal - DOE performed two separate hydraulic fracture experiments at the Baca power plant demonstration site. The 7-inch packer was set at about 915M (3000 ft).

where the formation is normally about 232C (450F). Two Otis packers with L'Garde Y267 EPDM elements were used, one for the fracture treatment and then one for drill stem tests. This procedure was successfully performed in March and then again in October of 1981.

Normally for stimulations with temperatures above 149-177C (300-350F), Republic Geothermal takes special precautions to protect marginal seal elements made from standard elastomer compounds. They first cool the well, circulate ambient water while the packer is being run, leave the packer unset until the moment it is needed so that ambient water circulation can be continued, and then just prior to pumping the packer is set.

The above procedure required for packers with standard elastomer compounds is unnecessary for geothermal packers with Y267 EPDM elements deployed in wells even above 260C (500F). Having to keep the packer in the balancing act through this crucial period just prior to pumping is eliminated since the packer can be set as soon as it is run in, checked out for leakage, and then forgotten, thus freeing personnel to concentrate on other critical problems. In addition, if the standard elastomeric packers fail to pack-off just before pumping, expensive equipment must stand by until successful sealing is accomplished. Hypothetically, for the Baca case, if a standard elastomeric packer would have been run and if it had to be replaced, it would have cost about \$18,000 in standby time.

The Baca well was cooled somewhat when the stimulation packer was run since ambient water was injected to assure that the well would remain killed. Hence, the maximum temperature the first stimulation packer experienced was 160C (320F) during the 12 hours it was packed off. The maximum pressure during stimulation was 21.2 MPa (3080 psi). The drill stem test packer only saw 171C (340F) and 4.1 MPa (600 psi) because of the cooling from 8000 barrels of fracture fluid and it remained packed off for 18.5 hours. Conditions for the second stimulation were about the same.

A total of five packers were retrieved, as with the Bakersfield packer, on the first attempt with no problems whatsoever, textbook retrievals. All packer seals looked as new which was expected at these temperatures since this is well below the Y267 EPDM capability. However, these same conditions are a challenge for standard elastomer compounds and Republic commented that even with the added precautions taken with standard elastomers under these same conditions, they would expect the post-mortem seals to be extruded and/or cracked. Figure 2 shows the first stimulation packer after retrieval.

GEOTHERMAL HYDRAULIC FRACTURE PACKER

LANL is the prime contractor on a major DOE geothermal demonstration, the Hot Dry Rock (HDR) Project. LANL has shown feasibility of the concept at 200C (392F) and is currently preparing to demonstrate the concept with deeper wells and higher temperatures up to 320C (608F). Many elastomer problems existed at 200C and, hence, LANL is especially concerned about elastomers for the hotter wells.

The basic HDR concept is simple, i.e. to establish a large heat exchanger in an impermeable geothermal reservoir. The heat exchanger is established by drilling two neighboring wells, fracturing between them and injecting cold water in one well and producing hot water from the other. Figure 3 is a schematic of the HDR wells.

The wells have been drilled, and at this writing are being hydraulically fractured to effect the communication between the wells. The job is extremely difficult because of the high temperature, 320C (608F) at EE-2 bottomhole; and the 35° slant from vertical in the portion of the wells where fracture is required.

Several packers have been tried including an open-hole Lynes packer, a Baker 9-5/8 permanent casing packer, and an Otis 9-5/8 retrievable casing packer. The packers are being set at about 3450M (11,300 ft.) where the temperature is about 232C (450F). Currently the Otis packer with Y267 EPDM equivalent elements made by BJ-Hughes appears to be the best option for this "impossible" job and has been run four times in EE-2. On the first trip problems occurred on run-in at 335M (1,100 ft.) and the packer was tripped out with no damage to the EPDM element. The second packer was run in to 3455M (11,335 ft.) and appeared to be set, but with some question due to severe drag. Fresh water was pumped up to a maximum rate of 12 BPM with the surface pressure reaching a maximum of 46.9 MPa (6800 psi). A total of 5700 bbls was pumped in 9.5 hours. After more than 18.5 hours the packer was released and tripped out and half of the EPDM element was missing. It was obvious that the element was physically torn apart on retrieval and the elastomer properties were intact. The third packer was run in and set at 3444M (11,300 ft.). The fluid was pumped at a rate as high as 5 BPM with an associated pressure of 40 MPa (5800 psi). Because of other unrelated problems the packer was tripped out after having successfully sealed. The fourth was set at 3444M (11,300 ft.) and withstood 15 hours of pumping at 46.2 MPa (6700 psi). Subsequently it successfully withstood vent back of H₂O and CO₂ at 225 C (437F) for about seven days. Parts of the element were retrieved and it was obvious that there was no significant degradation of the elastomer.

Three out of four of the Otis packer runs resulted in successfully sealing under very difficult circumstances. Recovered portions of the seal elements show that the BJ-Hughes N22-2 (Y267 equivalent) elements did not suffer any significant degradation.

CEMENTING WIPER PLUG

LANL also spent significant effort on cementing wiper plugs. Poor cement jobs at Fenton Hill placed question on the performance of prior elastomeric cementing wipers. Through an extended effort with Dowell, LANL was able to procure Dowell wipers made from Y267 EPDM.

As a matter of course LANL first immersion tests wiper plugs in the autoclave. The autoclave was heated to 289C (552F) and 29.7 MPa (4310 psi) where it was held for 30 minutes, and then

let cool down. The autoclave and test specimen are at elevated temperature for about 12 hours. Changes in appearance and hardness are recorded.

LANL tested several wipers with different elastomers. All prior wipers showed some degradation in this test, with the elastomer on one disappearing completely. The Y267 wiper has been the exception; it was unaffected by the test. Post test it appeared as new, and the hardness did not change. Shore A hardness was measured at 10 different locations before and after the test.

At this writing, one Y267 EPDM Dowell cementing wiper plug was run at Fenton Hill. A scab liner was cemented into EE-2 at about 4389 M (14,400 ft.) where the BHCT was 275C (525F). The cement job was successful, however, during drill-out of the cement the plug was not located and hard cement was tagged 21 M (70 ft.) below the wiper plug catcher. Therefore, this field use is termed inconclusive.

FLOAT VALVE

One major problem area encountered at Fenton Hill, New Mexico, was due to the failure of seals used on near-bit float (check) valves. Without the float valve, flowback can occur when a joint or stand of pipe is added to the string. This is due to the excess weight of the cuttings carried in the drill pipe-to wellbore annulus caused by the limited cuttings-carrying capacity of the clear-water drilling-fluid system. Flowback often plugged collars and downhole motors.

A solution to the float-valve-seal problem was initiated during the drilling of EE-2. The float-valve supplier (Bakerline, San Antonio, Texas) was contacted and initiated a development to replace the standard elastomeric seals with a high-temperature material.

Given information on the Y267 EPDM several elastomer suppliers provided several candidates. One, an EPDM formulation, provided sufficiently improved capability to accomplish the drilling. The formulation is not known to the authors nor the extent of the use of Y267 EPDM technology.

LANL first performed autoclave tests of the unknown EPDM seal in water at 280C (536F) for 30 hours. Softening of the seals was observed which implies that it is not equivalent to Y267 EPDM technology, but it did maintain its integrity. Y267 EPDM has been observed to maintain its properties for five days at the extreme temperature of 300C (575F). But nevertheless, the unknown EPDM was a major improvement over the prior three candidates that were run in the autoclave, one completely disappeared.

The unknown EPDM was subsequently successfully run on 30 to 40 hour rotary drill, and three to five hour turbodrill runs though the seals softened. The static formation temperature was 304C (580F) and the drilling fluid entered the string at 20C (68F) so the float valve was operating somewhere between these temperatures.

Significant improvements were made from a float valve which failed to one which successfully

operated. The data show that additional capability is available through use of seals using Y267 EPDM technology as opposed to the unknown EPDM formulation.

LOGGING TOOL SEALS

LANL had numerous elastomers problems with logging equipment on the earlier 200C (392F) wells at Fenton Hill. Hence, there was increased concern when faced with the challenge of deeper, hotter wells anticipated to be 275C (525F) but which ended up to be 320C (608F). Consequently, a program was planned to address this problem using Y267 technology.

L'Garde fabricated O-rings and a cable bend protector from the Y267 EPDM packer seals compound for the LANL cablehead. LANL tested cablehead O-rings in an autoclave with water and Mobil One Oil. 24-hour cycles were run with the temperature and pressure on for eight hours and off for 16, simulating tripping in and out of the hole. The best prior performance achieved was with commercial fluoroelastomer O-rings which literally disintegrated after one cycle.

LANL ran the Y267 EPDM in water for five cycles or five days before stopping the test to examine the seals. This included a 24-hour run on the fifth day which provided a total test time of about 56 hours for this one seal. The temperature was nominally 275C (525F) and the pressure was nominally 51.7 MPa (7500 psi). The O-ring looked excellent after this test and obviously could have continued further cycling.

They also ran the Y267 EPDM in Mobil One Oil for four cycles or four days and stopped the tests to examine the seals. The temperature was nominally 275C (525F) with a one hour excursion to 380C (716F) during one of the 24-hour cycles. LANL estimates that the seal was exposed to approximately 340C (633F). The differential pressure was nominally 51.7 MPa (7500 psi). The seals looked good and were sealing after four cycles, although there was some swelling from the high-temperature oil and some permanent deformation as would be expected for these conditions. They hope to go ten trips operationally before they must change out the O-rings.

These tests are landmark tests because this is the first time the Y267 EPDM was tested to the 51.7 MPa (7500 psi) differential pressure range, the first time the Y267 EPDM was tested in 100% oil, the first time the Y267 EPDM was tested in oil at extreme temperatures (excursions to 340C (644 F)), and the first time the Y267 EPDM was subjected to a cycling environment. The above results are even further underlined when considering the fact that EPDMs are generally not used in oil environments because they are extremely vulnerable to degradation and swelling. These tests at the extremely high temperatures provide substantiating evidence that the Y267 EPDM is extremely serviceable in oil.

Subsequent to the autoclave testing, LANL equipped their high-temperature cablehead with L'Garde Y267 EPDM O-rings and cable bend protector. The cablehead was run with a temperature probe

into the new EE-2 well which is about 4600M (15,000 ft.) deep with a bottomhole temperature of 320C (608F). The longest service experienced by Y267 EPDM parts at this writing is bottomhole for ten hours and above 200C (400F) for 15 hours. The parts sealed and looked excellent after this set of round trips with the most major problem being only some permanent deformation of the O-rings. LANL plans to try for as many as ten trips on a single set of elastomeric parts when the opportunity avails itself. They have now converted the seals for all logging tools over to Y267 EPDM.

LANL¹⁰ ran some temperature surveys for Union at the Baca using their cablehead equipped with Y267 EPDM seals. The well temperature was approximately 300C (575F) and the Y267 EPDM parts were exposed for a maximum of four to six hours. The rubber parts were changed whenever the cablehead was dismantled for other reasons. The seals performed entirely successfully.

MISCELLANEOUS LAB/FIELD TESTS

Battelle Northwest¹¹ ran into rubber seal problems on an inline corrosion probe they are testing. They ran autoclave tests in 200C (400F) brine on the Y267 EPDM prior to installing it in the field. They have tested several compounds and confirm that the Y267 EPDM stands up better than any other elastomer they have tested. They subsequently ran excellent comparative tests at the Magma Plant in the Imperial Valley. A test loop is available which parallels the plant where several instruments can be tested simultaneously. Battelle ran corrosion probes with fluoroelastomer (FKM), perfluoroelastomer (FFKM), and Y267 EPDM seals side-by-side. Both other seals failed within 2 weeks; the FKM became brittle, and the FFKM cracked and split. The Y267 EPDM was still running after eight months when the plant was shut down. This is the longest performance logged for this application for any elastomeric seal and Battelle concludes that Y267 EPDM is the only elastomer they know of which works for this application.

LANL/Union Oil¹² ran an explosive stimulation experiment at the Geysers. They encountered seal problems during preliminary hot water tests with their timer/detonator vessel which is immersed in the explosive during operation. Immersion tests were subsequently run in the explosive at 246C (475F) and 6.9 MPa (1000 psi) with four different elastomer compounds and the Y267 EPDM was clearly the best performer. Tests were also successfully rerun in 260C (500F) water in an autoclave. Y267 EPDM seals were ultimately installed in both pressure vessels; the timer/detonator, and the overall container. The timer was set for 48 hours for the downhole tests and the detonation occurred at the second implying that the seals must have performed flawlessly.

PRECISION RUBBER ASSOCIATIONS¹³

Precision Rubber provided Y267 EPDM technology elastomeric seals to two of their customers who reported notable success. One was the WKM Division of ACF Industries and the second was Baker Service Tools.

WKM reported that application of the Y267 EPDM in their 3-inch valve removal tool has been

a complete success. They tested several EPDM and AFLAS compounds from different vendors in their lab with 260C (500F) saturated steam. They reported that deterioration was least with Y267. They performed a perfect demonstration for personnel from four operating companies at the Geysers in 229C (445) geothermal steam. The same seal has been used as many as three times with potential for more. Valve changing on a live wellhead has historically been a safety and noise problem at the Geysers.

Baker Service Tools reported successful use of Y267 EPDM O-rings in a high-temperature, wireline setting tools powered by gas generator charges. Lab tests were extreme with pressures up to 207 MPa (30,000 psi) at 316C (600F) in water, motor oil, and synthetic oil. Static and dynamic leakage was too slight to measure. As of August 1982 there were five applications in deep, hot, gas wells with a perfect record of no Y267 EPDM failures in the field or laboratory.

PARKER SEAL ASSOCIATIONS²

Parker Seal customers designated as #1 and #2 have done thorough laboratory testing which is preliminary to field applications. In both cases, Parker's E962-85 (modified Y267) was compared in the laboratory against multiple other compounds in 300C (575F) steam and was rated as number one. Customer #1 repeated his laboratory tests with the same conclusion the second time. Customer #1 has fielded his hardware in 300C geothermal wells and successfully survived multiple applications of the same set of seals.

Customer #2 is planning to field E962-85 seals in 27 of 28 seals in a Gulf Coast gas well.

BJ-HUGHES ASSOCIATION¹⁴

BJ-Hughes customer, Otis Engineering Corp., has internal seals in their 9-5/8 packer described above. These are sliding seals for an internal expansion joint or "slick" joint for the attached tubing. Lab tests were successfully run by Otis at 275C (525F) with a 20.7 MPa (3000 psi) differential for ten days. The diametral gap was substantial at .508 mm (.020 in.). The slick joint seals were successfully fielded at Fenton Hill in the #4 packer described above. The tubing was successfully stabbed in seven times where the temperature was 232C (400F) and the well was inclined 35° from the vertical.

MISCELLANEOUS LAB TESTS

TerraTek ran drill bit seal tests on several elastomers including Y267 EPDM. The test exposes the seal to grease on one side and rock cuttings, sand, and water on the other. The specimen is raised to a presoak temperature to simulate tripping into the well, cooled to 150C (302F) to simulate introduction of cold drilling fluid, and then the dynamic test is initiated. The test provides a rotating motion and on each cycle radial and longitudinal jogs are super-imposed on the motion. At the time they ran Y267 EPDM, a 40 to 50 hour run was quite respectable. With the Y267 a presoak temperature of 288C (550F) was chosen; this was a first for this high a test temperature. Both

TerraTek and L'Garde were pleasantly surprised when the Y267 EPDM was still sealing after 104 hours. TerraTek remarked that the Y267 appeared excellent after the test,¹⁵ less degraded than all other elastomers tested.

Parker Seals has run extensive lab tests on their modified Y267 EPDM, E962-85. Of particular interest are tests in a 5% NACE A solution, an aqueous amine solution. The tests were run for 168 hours at 100C (212F). The E962-85 was compared against four other elastomers with very favorable results. It experienced 16.2% compression set while the other compounds experienced from 30 to 80%.

Sperry Vickers¹⁶ ran long-term compatibility tests of elastomers in an aqueous solution of hydrazine at 218C (425F). They saw no change in the Y267 after 85 days.

AVAILABILITY OF Y267 EPDM TECHNOLOGY

A major objective of the DOE effort was to assure that the developed technology is available for widespread field use. To this end, L'Garde was contracted to transfer the technology. After an application and careful evaluation process, three companies were selected for transfer. The transfer is now complete and molded parts based on Y267 EPDM technology are available in commercial quantities from BJ-Hughes Rubber, Parker Seal, and Precision Rubber. The full spectrum of large and small parts are available from these suppliers. In addition, the technology is available for R&D efforts directly from L'Garde.

CONCLUSION

A broad spectrum of applications of Y267 EPDM technology are presented. Over 15 laboratory and over 20 field trials are documented providing convincing proof that Y267 EPDM technology is at the cutting edge. The data indicate that Y267 EPDM is the superior elastomer for high temperature aqueous reducing environments. Test data is also building which indicates superiority for high-temperature hydrocarbon reducing environments. There is one environment where Y267 EPDM is not a good choice, where there is free access to air or oxygen at high temperatures. Specific details need to be carefully considered under these circumstances as to applicability.

In addition the Y267 EPDM technology is out of the development laboratory and is presently available in commercial quantities and for R&D situations from described sources.

ACKNOWLEDGEMENTS

The effort represented by this paper was sponsored by the United States Department of Energy, Division of Geothermal Energy. Mr. J. V. Walker and Dr. R. R. Reeber were instrumental in providing the opportunity to engage in this research and development, and provided valuable guidance throughout the effort. In addition, Dr. P. E. Cassidy of Texas Research Institute contributed significantly as technical coordinator for DOE as did Mr. A. J. Adduci of the San Francisco office of DOE as the technical administrator. Dr. L. E. Kukacka

of Brookhaven National Laboratory provided valuable guidance on the Technology Transfer to industry and the Binary Plant Compatibility Testing Program.

Invaluable information was obtained through the cooperation of several Government laboratories and commercial companies in the oil and gas, geothermal, and chemical products industries. Mr. J. K. Sieron of the Air Force Materials Laboratory was especially helpful.

REFERENCES

1. Taylor, D.: Private Communication, Otis Engineering Corp., Dallas, TX.
2. Barsoumian, J.L.: "Improved Energy Sealing Capability," presented at DOE Geothermal Engineering and Materials (GEM) Program Conference San Diego (6-8 October 1982).
3. Hirasuna, A. R. et al.: "Geothermal Elastomeric Materials (GEM) Program - Final Report for Period 1 October 1976-30 June 1979," L'Garde, Inc., Newport Beach, CA SAN/1308-2, U.S. Department of Energy - Division of Geothermal Energy (July 1979).
4. Hirasuna, A. R. and Stephens, C. A.: "Development of Elastomer Compounds for High-Temperature Service," NACE Material Performance (December 1981) 52-58.
5. Hirasuna, A. R., Friese, G.J. and Stephens, C.A.: "High-Temperature Y267 EPDM Elastomer," Geothermal Resources Council Bulletin (March 1982) 3-8.
6. Friese, G. J.: "Short and Long-Term Tests of Elastomers With Hot Hostile Fluids," L'Garde, Inc., Newport Beach, CA; Brookhaven National Laboratory/U.S. Department of Energy-Division of Geothermal Energy (to be published).
7. Bostock, J.: Otis Test Report Number 1395-31982 (19 March 1982).
8. Verity, R.: Private Communication, Republic Geothermal, Santa Fe Springs, CA.
9. Archuleta, J. and Dennis, B.: Private Communication, Los Alamos National Laboratory, Los Alamos, NM.
10. Dennis, B. and Todd, B.: Private Communication, Los Alamos National Laboratory, Los Alamos, NM.
11. Shannon, D. and Danielson, M.: Private Communication, Battelle Northwest, Richland, WN.
12. Allen, C.: Private Communication, Union Oil Research, Brea, CA.
13. Gillette, H.: "A Fabrication Report on L'Garde Y267 Geothermal Compound," Presented at DOE Geothermal Engineering and Materials (GEM) Program Conference, San Diego, (6-8 October 1982).
14. Logsdon, L.R. and Long, W.C.: "Elastomers for Service in Deep Well Environments," Presented at DOE Geothermal Engineering and Materials (GEM) Program Conference, San Diego (6-8 October 1982).

15. Hendrickson, R. and Winzenreid, R.: Private Communication, TerraTek, Salt Lake City.

16. Nowicki, D.: Private Communication, Sperry Vickers, Jackson, MS.

TABLE I²
AGEING IN STEAM BY PARKER SEAL

TEST	PARKER E692-75 ^a	PARKER E962-85 ^b
<u>168 Hours @ 288C (550F)</u>		
Hardness, Type A, PTS (Chg., PTS)	67(-7)	82(-5)
Tensile Strength, PSI (Chg., %)	1330(-33)	1740(-19)
Elongation, % (Chg., %)	261(+47)	120(+6)
Modulus @ 100%, PSI (Chg., %)	376(-50)	1710 --
Volume Change, %	-5.3	+2.1
Compression Set, %	85.7	46.7
<u>168 Hours @ 316C (600F)</u>		
Hardness, Type A, PTS (Chg., PTS)	Deteriorated	65(-22)
Tensile Strength, PSI (Chg., %)	Deteriorated	506(-76)
Elongation, % (Chg., %)	Deteriorated	109(+14)
Modulus @ 100%, PSI (Chg., %)	Deteriorated	466 --
Volume Change, %	Deteriorated	+4.0
Compression Set, %	Deteriorated	85.7

a. Best prior generation EPDM as tested by L'Garde.

b. Based on Y267 EPDM Technology.

TABLE II
SUMMARY OF Y267 EPDM LABORATORY & FIELD TESTS

LABORATORY									FIELD								
TEST	FLUID	TEMP		AP		TIME			TEST	FLUID	TEMP		AP		TIME		
		C	F	MPa	psi	HRS.	DAYS	MOS.			C	F	MPa	psi	HRS.	DAYS	MOS.
L'GARDE ECT IMMERSION ⁶	Brine, C ₄ H ₁₀ , Oil	191	375	N/A			180	6									
L'GARDE ECT O-RINGS ⁶	Brine, C ₄ H ₁₀ , Oil	266	510	20.7	3000		5										
OTIS PACKER ¹	H ₂ O	300	575	20.7	3000		5		BAKERSFIELD STIM ¹	Steam + Crude	204	400	1.7	250		150	5
OTIS PACKER ⁷	Swt. & Sour Crude	232	450	138	20,000		5		BACA GEOTHER FRACT (2x) ⁸	H ₂ O + Fract Fluid	160	320	20.7	3080	12	.5	
									BACA GEOTHER DST (3x) ⁸	H ₂ O	171	340	4.1	600	18.5	.8	
									FENTON HILL GEO FRACT (4x) ^d	H ₂ O, CO ₂ , H ₂ S	232	450	49.7	7200	180	7.5	
LANL, CEMENT WIPER	H ₂ O	289	552	29.7	4310	21.5	.9		FENTON HILL CEMENT	Cement/H ₂ O	275	525	0	0	.5	.02	
LANL, FLOAT VALVE ^e	H ₂ O	280	536	0	0	30	1.3		FENTON HILL DRILLING ^e	H ₂ O	?	?	13.8	2000	40	1.7	
LANL O-RING TESTS ^{3, 10}	H ₂ O, Mobil 1	275	525	51.7	7500	40	1.7		FENTON HILL LOGGING ^{a, 10}	H ₂ O, CO ₂ , H ₂ S	317	603	41.4	6000	10	.4	
									BACA LOGGING ^{a, 10}	H ₂ O	300	575	11.7	1700	6	.3	
BATTELLE RICHLAND ¹¹	H ₂ O	204	400	4.1	600		4		MAGMA CORROSION PROBE ¹¹	Brine	180	356	N/A	N/A		240+	8+
UNION/PI EXPLOSIVE ¹²	Explosive	246	475	6.9	1000	24	1		GEYSERS EXPLOSION EXPT ¹²	Steam	260	500	4.6	665	48	2	
WKM VALVE CHANGER ^{b, 13}	Steam	260	500	4.8	700	1	.04		GEYSERS VALVE CHANGER (3x) ^{a, 13}	Steam	260	500	2.8	400	.75	.03	
BAKER SERVICE SETTING TOOL ^{b, 13}	Steam, Oil	316	600	207	30,000	24	1		GULF COAST GAS WELLS (5x) ¹³	Gas Wells	?	?	?	?		?	
CUSTOMER #1 (2x) ^c	Steam	300	575	?	?		?		GEOTHERMAL COMPLETION ^{a, 2}	?	300	575	?	?		?	
CUSTOMER #2 ^c	Steam	300	575	?	?		?		GULF COAST, 27/28 SEALS TO GO ²	Gas Wells	?	?	?	?		?	
OTIS SLICK JT ^{d, 14}	H ₂ O	275	525	20.7	3000		10		SEVEN STAB-INS, GEOTHERMAL ¹⁴	H ₂ O	232	450	49.7	7200	180	7.5	
TERRATEK DRILL BIT SEAL ¹⁵	Grease/Debris, H ₂ O	288	550	0/1.4	0/200	104	4.3		a. The same set of seals were used more than once in these applications. b. Precision Rubber Associations. c. Parker Seal Associations. d. BJ-Hughes Associations. e. EPDM formulation unknown, degradation in testing apparent.								
PARKER ²	Amines, NACE A	100	212	N/A	N/A	168	7										
SPERRY VICKERS ¹⁶	HYDRAZINE	218	425	N/A	N/A		85										

TABLE III
SUMMARY MATRIX
BINARY POWER PLANT COMPATIBILITY TESTING

Number Tested	Immersion (6 Fluids)			O-Ring (3 Fluids)	
	5D	2 mos.	6 mos.	2D	6 mos.
34 Compounds 191C (375F)	X				
8 Selected 191C (375F) 232C (450F) 266C (510F)		X	X	X X X	
4 Selected 204C (400F)					X

Compound	Rank	Temperature		
		191C(375F)	232C(450F)	266C(510F)
L'Garde EPDM Y267	1		Brine
	1		Isobutane
	1		ASTM #1 Oil
Parker EPDM E692-75	1
	2
	2
L'Garde Viton 501	4		Brine
	5		Isobutane
	3		ASTM #1 Oil
Vernay Lab Fluorel VL1503M3	7
	4
	3
Precision Fluorel 16959	8		Brine
	3		Isobutane
	5		ASTM #1 Oil
Seals Eastern AFLAS 7170X14	3
	7
	6
L'Garde AFLAS 291	4		Brine
	5		Isobutane
	8		ASTM #1 Oil
DuPont Kalrez 1018	4
	7
	7

Damage limited to moderate set and swelling.

Severe set, cracks or splits, signs of extrusion.

Nibbling or catastrophic failure.

Fig. 1—46-hour O-ring test results, 20.7 MPa (3000 psi) ΔP.

Fig. 3—Schematic of Fenton Hill HDR wells.

Fig. 2—Post-mortem packer with Y267 EPDM seal after geothermal hydraulic fracture. Seal is like new.