ELASTOMER LINERS FOR GEOTHERMAL TUBULARS
Y267 EPDM LINER PROGRAM
FINAL REPORT

BNL--40946

DE88 007014

LTR-86-AH-127
DECEMBER 1986

A.R. HIRASUNA
D.L. DAVIS
J.E. FLICKINGER
C.A. STEPHENS

L'GARDE, INC.
15181 WOODLAWN AVENUE
TUSTIN, CA 92680

PREPARED FOR

BROOKHAVEN NATIONAL LABORATORY
ASSOCIATED UNIVERSITIES, INC.
UPTON, L.I., NEW YORK 11973
SUBCONTRACT NO. 186211-S

DISCLAIMER

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FORWARD

The technical information reported herein was developed by L'Garde, Inc., Tustin, CA, and Unocal Science and Technology Division, Brea, CA under the Y267 EPDM Liner Program, BNL Subcontract No. 186211-S.

The work was sponsored by DOE/San Francisco Operations with cost sharing by Unocal Geothermal Division through Brookhaven National Laboratory. The program was managed for BNL by Dr. L.E. Kukacka with DOE cognizance by Messrs. A.J. Adduci, W. Bryan, G. Petersen, and Dr. M. Malloy at San Francisco Operations and Mr. R. LaSala at Washington, D.C. Headquarters. Dr. W.C. Allen was liaison for Unocal.

The effort was directed for L'Garde by Mr. A.R. Hirasuna. The development of this report and the technology advancement achieved under the Y267 EPDM Liner Program are also attributed to the following L'Garde personnel: Messrs. D.L. Davis, J.E. Flickinger, and C.A. Stephens.

In addition to his liaison tasks, Dr. W. C. Allen of Unocal also contributed directly to the technical effort. Other Unocal personnel in field operations carried out those essential tasks of planning, installation, surveillance, and removal of the liner prototype that made the field test possible.

STATEMENT OF OBJECTIVE

Investigate the feasibility of protectively lining steel geothermal tubulars with the Y267 EPDM elastomer.

ABSTRACT

The elastomer, Y267 EPDM, has been identified as a hydrothermally stable material which can operate at temperatures in excess of (320C) (608F). The goal of the Y267 Liner Program was to demonstrate the feasibility of using this material as a liner for mild steel tubulars to prevent or mitigate corrosion. If successful, the usage of EPDM lined pipe by the geothermal community may have a significant impact on operating costs and serve as a viable alternative to the use of alloyed tubulars.

Tooling procedures were developed under this program to mold a 0.64 cm (0.25") thick Y267 EPDM liner into a tubular test section 61 cm (2') in length and 19.1 cm (7.5") in diameter (ID). A successful effort was made to identify a potential coupling agent to be used to bond the elastomer to the steel tubular wall. This agent was found to withstand the processing conditions associated with curing the elastomer at 288C (550F) and to retain a significant level of adhesive strength following hydrothermal testing in a synthetic brine at 260C (500F) for a period of 166 hours. Adhesive strengths varied from a low of 3.4 (500) to a high of 6.2 MPa (900 psi) for cured samples. Following hydrothermal testing these strengths ranged from 0.07 (10) to 1.4 MPa (200 psi). These values reflect variations in the processing conditions associated with the molding and curing operations and the types of alloys tested. Bonding tests were conducted on specimens of mild carbon steel and several alloys including Hastelloy C-276.

An objective of the program was to field test the lined section of pipe mentioned above at a geothermal facility in the Imperial Valley. Though a test was conducted, problems encountered during the lining operation precluded an encouraging outcome. The results of the field demonstration were inconclusive.

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1.0 BACKGROUND, SUMMARY, AND RECOMMENDATIONS

This introductory section is designed to provide an executive synopsis of the Y267 Liner Program. It captures the overall essence of the entire Program by providing the background, summary of accomplishments, and recommendations.

1.1 BACKGROUND

Production of geothermal energy at resources similar to the Salton Sea area of the Imperial Valley in California presents its own unique set of engineering problems. The characteristic highly corrosive geothermal fluids render standard mild steel tubulars undesirable in many process areas of a geothermal plant.[1,2,3] Cheaper corrosion resistant alloys may also be undesirable in certain process areas because of their vulnerability to embrittlement, cracking, or pitting caused by the presence of chloride or hydrogen ions, hydrogen sulfide, and other environmental conditions.

More corrosion resistant alloys are a possible solution, however, they may be exorbitantly expensive. Standard 8-5/8 inch 36 ppf steel tubing costs 35.40 $/m (10.80 $/ft) while the equivalent Hastalloy C-276 tubing costs over 1,640 $/m (500 $/ft.). In addition, the supply of tubulars made from other than steel tubing is limited and the problems of availability and lead time are ever present.

The above scenario naturally leads to a next question, i.e., are there any less expensive more practical alternatives? When addressing that question for Salton Sea geothermal tubulars, the answer is possibly yes. Y267 EPDM is an elastomer compound developed by DOE/L'Garde, Inc. for high temperature geothermal seals. Y267 has proven to be eminently successful and works better than any other elastomer known for geothermal applications.[4,5] The success of Y267 EPDM as a geothermal seal material leads to the possibility of using it as a protective liner for tubulars. Cement or elastomeric piping liners have been used for decades in the chemical processing industry for relatively benign temperatures. Hence, the concept is well proven; the uncertainty deals with the much more hostile geothermal environment. Assessing the applicability of Y267 EPDM as a tubular liner in the hostile geothermal environment is the objective of the Y267 Liner Program.

Unocal was a prime mover on this liner research, initiating a small effort with L'Garde several months prior. The planning of this Program was a cooperative effort between Unocal, Brookhaven National Laboratory (BNL), and the U.S. Department of Energy - San Francisco Operations (DOE-SAN).

This planning ultimately resulted in the Y267 Liner Program, BNL Subcontract No. 186211-S, with Unocal participating as a cost sharing participant.

1.2 SUMMARY

The Y267 EPDM Liner Program was originally planned in early 1984. At that time, the DOE geothermal materials R&D budget was very limited, hence, a "bare-bones" minimum cost development program was configured. The defined objective was to assess the fundamental feasibility of Y267 EPDM lined steel tubulars, excluding interfaces at the ends of the tubular joints where flanged or screwed connections exist. The Program was structured with an optional task to apply L'Garde's Series 600 elastomer/steel bonding or coupling system which would only become necessary if a commercial alternative failed. Another cost expediency was to optimistically assume that no problems would arise when scaling-up from laboratory coupons to a 91.4 cm (36 in) long tubing prototype for field testing. Most of the desired objectives were accomplished.

The objectives of the Y267 Liner Program were as follows:

- Evaluate the viability of Y267 EPDM as a geothermal corrosion protection lining.
- Evaluate feasibility of lining steel tubulars with Y267 EPDM.

The following were performed to accomplish those objectives:

- Assess the best commercially available elastomer/metal bonding system known for application to the Y267 Liner.
- If commercial system does not survive Y267 Liner temperatures, implement option to adapt Series 600 elastomer/metal bonding system.
- Design a Y267 lined tubular section for field prototype testing.
- Fabricate prototype tubing specimens for geothermal field test.
- Field test prototype in geothermal brine line.

A significant part of evaluating the viability of Y267 EPDM tubing liners was successfully demonstrated. Y267 EPDM liners were successfully molded into tubing prototypes, where a previous attempt by another effort were refined to the point where perfect liners could be molded into the 8-5/8 inch prototype tubing as shown in Figure 1.

The commercially available elastomer/metal coupling system could not survive the postcure of Y267 EPDM at 288C (550F) so the effort was shifted to the Series 600 Coupling System. The Series 600 Coupling System survived the cure and postcure very well with over 560% the adhesive strength of the commercial system and, in addition, maintained reasonable adhesive strength after 1 week of aging in synthetic geothermal brine at 260C (500F). However, though the Series 600 Coupling System worked very well on laboratory coupons, the results were unreliable when scaled up to field prototypes. Further development associated with adhesion to prototype tubulars was precluded by the scope of the program.

Even though the adhesion was not reliable, the prototype was modified such that it could be fielded in spite of the adhesion problems. It was reasoned that this expedient might produce some useful data versus the alternative of having no field data whatsoever.

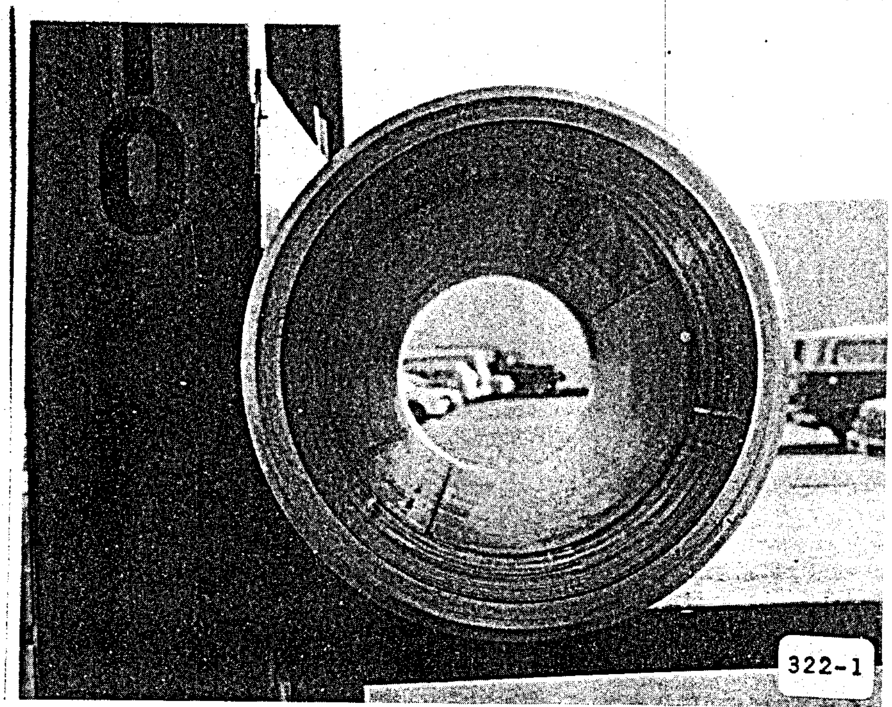
The modified prototype was fielded by Unocal in their Salton Sea geothermal field. Figure 2 shows the field prototype, shiny section on right, attached to the stinger. The entire assembly was inserted into the brine line and mounted in place at the end of the stinger. The prototype was installed in a surface supply line from a single well. Full flow passed through the prototype when operating. The temperature of the stream reached about 246C (475F).

The field test did not yield an evaluation of the viability of Y267 EPDM liners. In spite of the provisions to compensate for the poor adhesion on the tubing prototype, the Y267 liner was flowed out of the prototype and no conclusions as to the viability of Y267 EPDM lined tubulars in geothermal applications could be made.

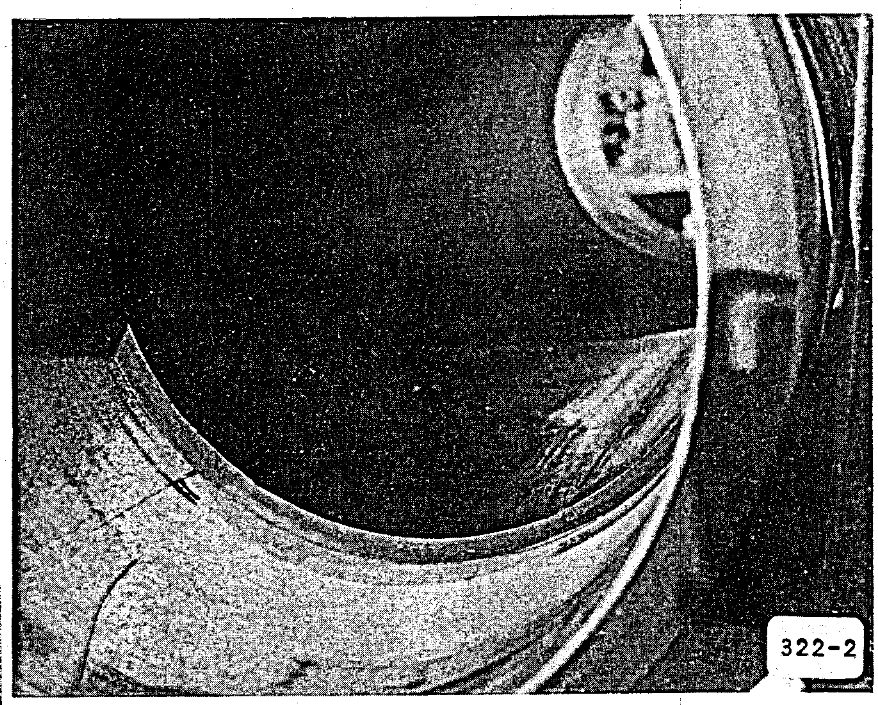
Fabricating and fielding the liner prototype yielded valuable experience. It pointed up the need for moderately good adhesion, verified the specimen design, and pointed up the need for some pressure gages or recorders to produce some diagnostic data. The experiment provided significant benefits for future field tests.

1.3 RECOMMENDATIONS

As described above, the development could not be carried to the level of completion and confidence desired. The overall recommendations include improving the Y267/metal bonding plus going beyond the present project to



Note flawless molding throughout. Two sets of double lines 180^{0} from each other are mold parting joints.



Note chamfered end which interfaces with metal ring to be inserted.

Figure 1. Y2677 EPDM Liner Prototype

complete full-scale demonstration of the liner concept for geothermal applications. This breaks down into the following detailed recommendations.

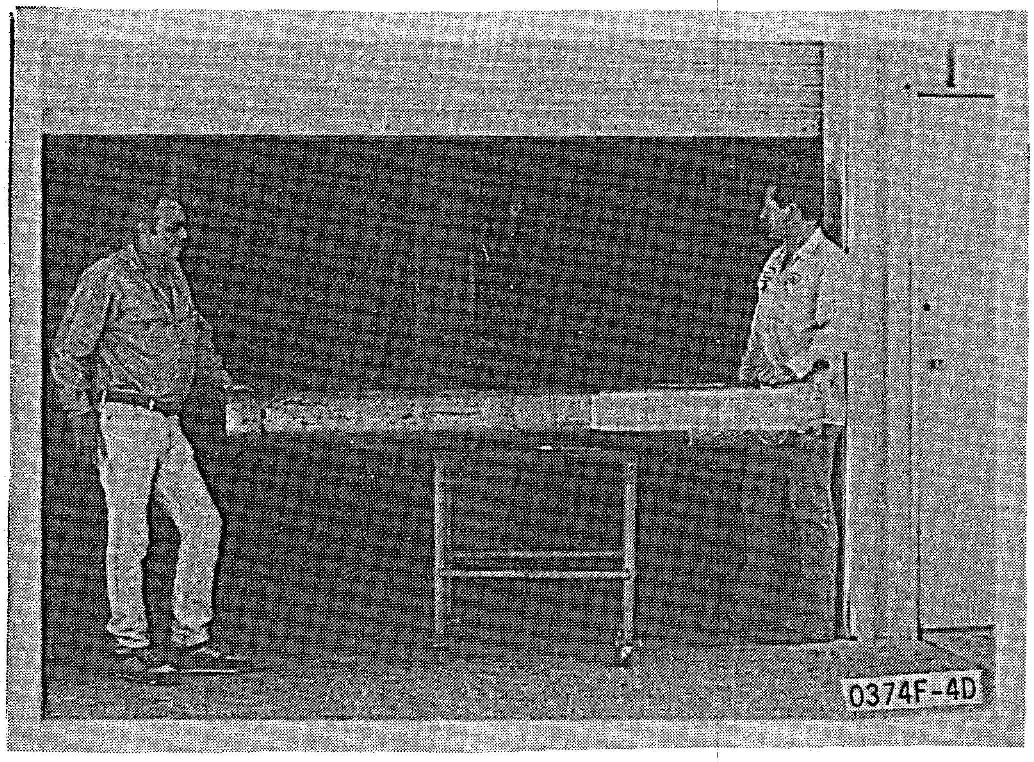


Figure 2. Pretest Field Prototype Mounted to Stinger

- Complete the scale-up of the coupling system from coupons to tubing such that reliable Y267/metal bonding results.
- Run another field test of a short prototype.
- Develop the end interfaces for the Y267 liner at the tubing joints.
- Scale-up the process for entire joints of tubing fabricated using production methods.
- Run a full-scale field test of an entire length of lined tubing with end interfaces.

2.0 COMMERCIAL COUPLING SYSTEM EVALUATION (TASK 2)

The objective of the Task 2 effort was to evaluate the best commercially available adhesive system known. The system was reported to have been successfully tested with Y267 EPDM, but at test temperatures below 260C (500F) and processing temperatures below 204C (400F). It cannot be emphasized too emphatically that the commercial system was not claimed to be able to survive the Y267 EPDM 288C (550F) postcure nor the requirements set for the liner. The purpose of this task was to evaluate the system for the liner application with the outside hope it might meet these very hostile requirements and quickly fulfill the coupling system needs for this program. This effort involved processing at standard temperatures as well as various reduced temperatures but times which provided an Arrhenius equivalent to the standard Y267 process, and immersion testing at 260C (500F) for 166 hours.

2.1 TESTS AND SPECIMENS

This section describes the various tests and test specimens employed.

2.1.1 Adhesion Test

Measuring adhesion of a low modulus material to a high modulus material can be complicated by various secondary phenomena. Often a 25.4 mm (one-inch) wide strip of the low modulus material is peeled from the high modulus material, and the force to do so provides the measure of the quality of the adhesion. The results can be misleading because the peeling stress for the same force depends on the modulus and thickness of the low modulus material. To avoid these interactions, L'Garde uses a test which provides a simple shear at the bond line.

The test specimen consisted of 15.9 mm (0.625 inch) diameter rod which was compression molded into the rubber for 51 mm (2 inches) of its length. Welding rod was used since it provided an inexpensive source of a wide variety of alloys with a minimum of preparation. Rods were cleaned by sanding and then washing with MEK. The rod was then pulled longitudinally out of the rubber with a tensile tester. The force required was the measure of the quality of the bond. The test was performed per ASTM D1871-68 method A.

2.1.2 Test Matrix

An evaluation matrix was designed which considers various parameters which include:

- Post cure temperature
- Synthetic brine
- Three metals

The synthetic geothermal brine used was formulated by L'Garde under the DOE Geothermal Elastomeric Materials Program in about 1975.[6] The formulation developed has become somewhat of a standard and has been used by others in the geothermal community. The constituency of the freshly prepared brine is as follows:

H_2S	300 ppm
CO_2	1,000
NaCl	25,000
H_2O	Balance

The actual test matrix is shown in Table I. The chemically aged specimens were aged in the above synthetic brine for 166 hours at 260C (500F).

TABLE I. EVALUATION TEST MATRIX FOR COMMERCIAL ADHESION SYSTEM

Post Cure Temperature	Rod-Pull				Coupon		Adhesive System
	Steel (RG 60)		C276		Alloy #1		
	V[a]	CA[b]	V	CA	V	CA	
288C (550F)	3 ea	3 ea	3 ea	3 ea			No (Control)
288C (550F)	3 ea	3 ea	3 ea	3 ea	1 ea	1 ea	Yes
260C (500F)	3 ea	3 ea	3 ea	3 ea	1 ea	1 ea	Yes
232C (450F)	3 ea	3 ea	3 ea	3 ea	1 ea	1 ea	Yes

[a]Virgin State
[b]Chemically Aged

Lower temperature postcures than are used for the Y267 process were investigated parametrically. This approach provided for the discovery of benefits of lower temperature processing if they existed. The postcure set-temperature histories are shown in Table II. Each postcure is calculated to be the Arrhenius equivalent of the 288C standard postcure (the first one listed in Table II).

TABLE II. ARRHENIUS EQUIVALENT POSTCURE SET TEMPERATURE HISTORIES

History Type	Maximum Post Cure Temperature	Time at Temperature, Hrs. 204C (400F)	232C (450F)	260C (500F)	288C (550F)
Standard	288C (550F)	1	1	1	5
Arrhenius	260C (500F)	1	1	39	-
Arrhenius	232C (450F)	1	297	-	-

Welding rod was used to provide the stock for the pull specimens. For ASTM A106 mild steel, RG 60 was used, and for the sample corrosion resistant alloy, Hastelloy C276 was used. The corrosion resistant alloy coupons representing a real field alternative were provided by Unocal. The test matrix included measurements of the cured specimens in the as-cured or virgin (V) state, and after being immersed or chem aged (CA) in synthetic geothermal brine for 166 hrs. at 260C (500F).

2.1.3 Test Specimens

Two types of test specimens were fabricated. The ASTM D1871 type pull test specimens provided quantitative data while the coupon specimens provided qualitative data only.

A. Modified ASTM D1871 Method A Test

The Method A specimen is basically a 50 x 200 x 12.5 mm (2 x 8 x 0.5 in.) slab of rubber with 15 rods molded through the middle of the thickness and perpendicular to the length. The rods used are 1.59 mm (0.0625 in.) in diameter, slightly larger than the 1.15 mm (0.045 in.) called out by D1871.

The 15 rod molding was cured and postcured as an integral unit. Rods number 1, 8, and 15 were not used in the testing but are included to facilitate the molding. RG 60 mild steel rods were positioned at 2, 3, 4 and 9, 10, 11 while Hastelloy C276 rods were positioned at 5, 6, 7 and 12, 13, 14. After postcure the molding was cut in half at position 8, and the

1-7 half was chem aged while the 9-15 half was retained in the virgin state. Figure 3 shows the general layout.

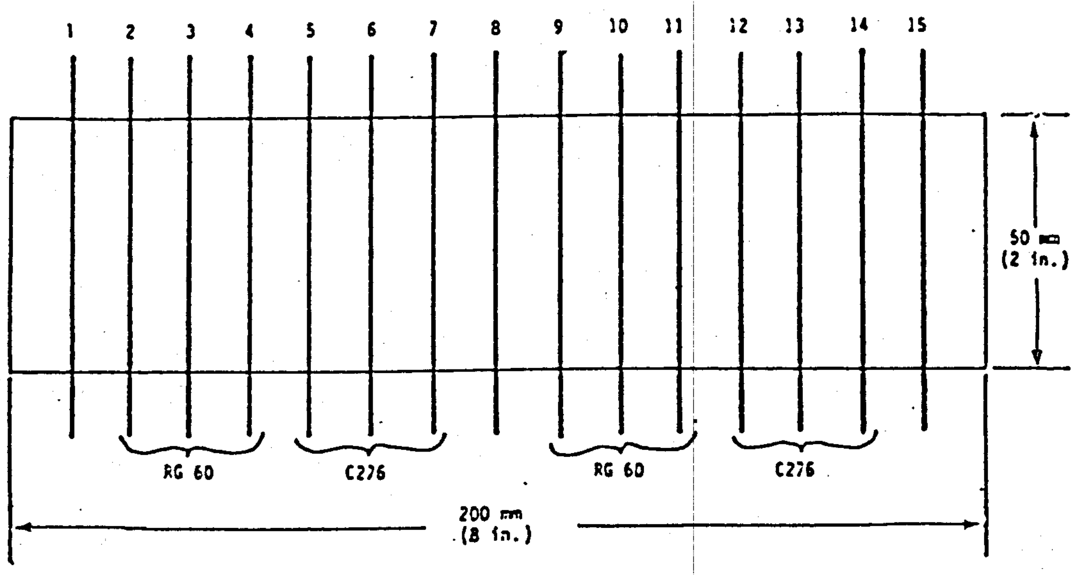


Figure 3. Rod Pull Test Specimen Lay-Out

B. Coupons

Six 25.4 x 76.2 x 3.18 mm (1 x 3 x 0.125 in.) #1 proprietary alloy coupons were provided by Unocal for evaluation. Two coupons were molded into 88.9 x 88.9 x 6.35 mm (3.5 x 3.5 x 0.25 in.) slabs and postcured. The slab was then cut in half and one coupon was chem aged while the other was retained in virgin state.

C. Adhesion System

All rods and coupons were prepared for molding by the commercial coupling system processor. They applied the adhesive system which currently works the best for them with Y267 EPDM. The metal parts were then molded by L'Garde into the two specimen configurations.

2.2 DISCUSSION OF RESULTS

Table III summarizes the results of all the evaluation testing of the commercial adhesion system. For each point on the pull-out force matrix, three redundant specimens were prepared and measured. The three measurements were subsequently arithmetically averaged and that value is shown in parentheses. Generally the adhesion of Y267 EPDM to the carbon

TABLE III. SUMMARY OF TEST RESULTS

Pull-Out Force

Postcure	RG60 Steel				Hast. C-276				Prop. Alloy #1	
	V[a] 9,10,11		CA[b] 2,3,4		V 12,13,14		CA 5,6,7		V	CA
	N	lb.	N	lb.	N	lb.	N	lb.		
288C (550F) Control No Adhes.	(85.8)	(19.3)[c]	(74.3)	(16.7)	(74.3)	(16.7)	(74.3)	(16.7)	---	---
	97.9	22.0	75.6	17.0	86.7	19.5	68.9	15.5		
	89.0	20.0	77.8	17.5	77.8	17.5	77.8	17.5		
	71.2	16.0	68.9	15.5	57.8	13.0	75.6	17.0		
288C (550F)	(142)	(32.0)	(113)	(25.3)	(48.9)	(11.0)	(108)	(24.3)	Bond	No Bond
	116	26.0	133	30.0	28.9	6.5	120	27.0		
	131	29.5	62.3	14.0	28.9	6.5	60	13.5		
	180	40.5	142	32.0	89.0	20.0	145	32.5		
260C (500F)	(161)	(36.2)	(117)	(26.3)	(40.0)	(9.0)	(45.4)	(10.2)	Bond	No Bond
	191	43.0	142	32.0	51.2	11.5	48.9	11.0		
	158	35.5	145	32.5	37.8	8.5	35.6	8.0		
	133	30.0	64.5	14.5	31.1	7.0	51.2	11.5		
232C (450F)	(208)	(46.8)	(69.8)	(15.7)	(72.5)	(16.3)	(41.4)	(9.3)	Bond	No Bond
	187	42.0	53.4	12.0	71.2	16.0	31.1	7.0		
	187	42.0	102	23.0	48.9	11.0	42.3	9.5		
	251	56.5	53.4	12.0	97.9	22.0	51.2	11.5		

[a] Virgin
[b] Chemically Aged
[c] Numbers in parentheses are averages of the three directly below

steel was better than to the Hastelloy C276. This probably results from one or a combination of two phenomena. First, the Y267 cure reaction products corroded the RG 60 steel and roughened the surface. Secondly the RG 60 steel is inherently more reactive than the Hastelloy C276, hence the probability of chemical bonding was higher. For both RG 60 and C276 there was not a lot of difference in their bond strength when compared to their control counterparts, with no coupling system. However, there was some general improvement on the RG 60. The bond shear strength was generally poor as 22N (50 lbs.) pull-out force corresponds to 0.876 MPa (127 psi). There was evidence of bonding to the proprietary alloy #1 in the virgin state, but none after chem aging.

The Table III coupon results are generally independent of the postcure conditions. On all virgin coupons there is evidence of adhesion, and on all chem aged specimens there is not.

In addition to the results shown on Table III, measurements were made on a rubber adhesion system combination which is known to work. L'Garde tested a natural rubber compound (456) being developed on a DoD program in conjunction with a Chemlok 205/233 adhesion system. Six pulls were made with an average pull-out force of 947N (213 lbs.) with a range of 845-1045N (190-235 lbs.). 947N (213 lbs.) corresponds to a shear stress of 3.74 MPa (542 psi).

2.3 CONCLUSIONS REGARDING THE COMMERCIAL COUPLING SYSTEM

The pull-out force data suggested the possibility of various correlations and interactions. It seemed apparent that the Y267 generally adhered somewhat better to RG 60 steel than the Hastelloy C276. There was also general evidence of Y267 bonding to the proprietary alloy #1 coupons prior to chemical aging but the bond was apparently thermally and/or hydrolytically destroyed in the autoclave aging.

Standing back and viewing these data on the commercial coupling system in an overall perspective, the following points predominated:

- The adhesion was generally poor, in the 89 to 222N (20 to 50 lb.) range compared to much better adhesion demonstrated by the 456 natural rubber/Chemlok system which was in the 845-1045N (190-235 lbs.) range.

- No adhesion remained on the #1 proprietary alloy coupons after chemical aging in synthetic geothermal brine for 166 hours at 260C (500F).

In addition, when chemical adhesion is low or nonexistent, the rod pull-out test must be carefully interpreted because it inherently provides the fullest advantage of any mechanical adhesion. Where a flat peel specimen may virtually fall apart, the rod pull-out specimen is forced to shear, and measurable shear stress may result due to friction.

Based on the above data, it was concluded that the commercial adhesion system for Y267 EPDM was not adequate for 260C (500F) tubing liner applications in geothermal brine. It was recommended that other means to secure the Y267 EPDM to the tubing be pursued.

3.0 ADAPTATION OF THE SERIES 600 COUPLING SYSTEM (TASK 3)

When Task 3 was initiated there was no known coupling system which could survive the Y267 EPDM 288C (550F) postcure, let alone survive the geothermal environment. The Series 600 Coupling System was shown to survive the postcure well, and furthermore to successfully survive autoclave ageing in synthetic geothermal brine with significant remaining strength.

3.1 TASK 3 BACKGROUND

From the outset of the Y267 EPDM Liner Program, the coupling or bonding of the Y267 liner to the metal tubular was known to be critical and anticipated to be a potential problem. Therefore, a two pronged approach was taken where two candidates could be considered. It was most desirable that the commercial coupling system work because that would be the quickest and least expensive solution to the problem. However, it was known that the commercial system was not claimed or not proven to survive the Y267 processing at 288C (550F) and the Y267 Liner operating specification, so a real question as to its adequacy existed.

The overall Y267 Liner Program was structured to quickly evaluate the commercial coupling system, and if it was adequate it was to be used for the liner prototypes for the field tests. If it appeared inadequate, BNL would exercise the option to have L'Garde adapt the Series 600 coupling system to the Y267 Liner which BNL eventually did, and Task 3 became a part of the Program.

3.2 ADAPTATION OF SERIES 600 COUPLING SYSTEM

The Series 600 coupling system enjoyed an excellent record of reliability for other applications at moderate temperatures in production run quantities with other elastomers. It was known that the system was a good candidate for the Y267 Liner application at geothermal temperatures, but it needed to be adapted and proven.

3.2.1 <u>Laboratory Specimen Testing</u>

An important aspect of the adaptation process was defining a specimen test which was economically acceptable and tested the critical characteristics of the operational situation.

At the time Task 3 began, the rod-pull test described in paragraph 2.1.3 existed and was operational. It alleviated the error introduced by peel-type tests where the stress at the peel failure point is influenced by the modulus and/or thickness of the material as discussed in Section 2.1.1. Though the rod-pull test provided a good means for quick evaluation of adhesion, it did not lend itself to detailed refinement of a coupling system. The bondlines are disturbed when the rod is pulled-out. In search of a better evaluation test, a butt-joint test where the joint is failed in tension was also used.

Figure 4 shows a schematic of the 20.3 x 25.4 x 31.8 mm (0.8 x 1.0 x 0.125 inch) butt-joint coupons as they were laid out in the mold. A standard ASTM slab mold was modified to accept 10 metal coupons to which the Y267 EPDM was molded. The hole in the coupon fitted over indexing pegs which hold the coupons in place. The elastomer was simultaneously molded to each of the 10 coupons around three of the four sides of each coupon. The entire set was removed from the mold and run through the Y267 EPDM postcure. Then each coupon was cut free into individual test specimens with the adhered tab of rubber as indicated by the dotted lines. The rubber was cut free on the side faces such that the tab was only adhered to the front face. Each specimen could then be tensile tested with the metal

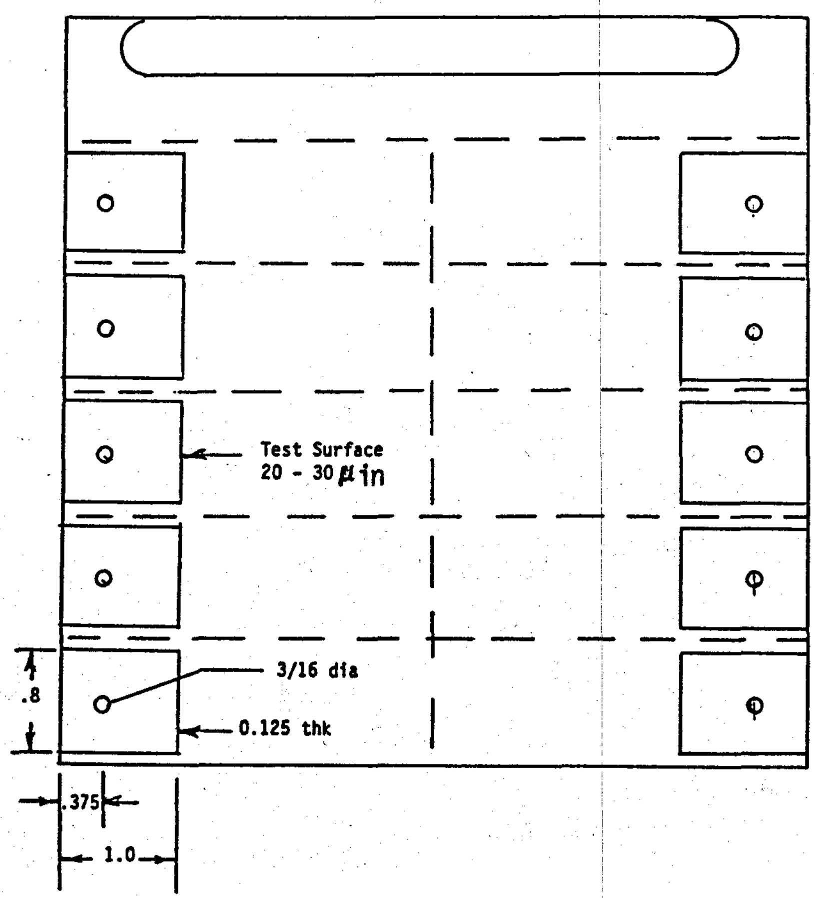


Figure 4. Butt-Joint Adhesion Test Schematic

coupon in one jaw of the tester and the Y267 tab in the other. The adhesion butt-joint was failed in tension at a grip separation rate of 50.8 mm/min, (2 in/min). The peak tensile stress calculated indicated the quality of the bonding.

Both the rod-pull and butt-joint adhesion tests proved to have a valuable role. The rods require no special preparation, they are simply cut from rod stock, hence provide an extremely inexpensive means for ballpark quantitative measurements. The butt-joint, though also inexpensive, requires more expensive machined coupons. With the rod-pull specimens, the elastomer protects the bondline from the brine in the autoclave except for one end, while with the butt-joint specimens, the entire four edges of the bondline are exposed. The butt-joints provide extremely good visual access to the failure interface after testing which was not disturbed by the wiping action as with the rod-pull; and a more accurate measure of the adhesion because they do not have a friction component.

3.2.2 Series 600 Coupling System Experimental Results

The Y267 Liner requirements to operate in brine at 260C (500F) imposed a significant challenge to advance the state-of-the-art for metal/elastomer coupling systems. There was no known system capable of meeting this high temperature in brine for production periods of time, or even the Y267 EPDM processing at 288C (550F).

Once the option for Task 3 was exercised, L'Garde launched into an effort which first confirmed the Series 600 Coupling System survived the 288C processing. Table IV shows the rod-pull data measured on the Series 600 Coupling System during this effort.

The purpose of Table IV was to indiscriminently show steel rod test data associated with each shop order (request to laboratory for work) such that the reader can see the substantial consistent advantage of the Series 600 Coupling System especially in the virgin state. Shop Orders 261 and 263 use the same coupling materials and process as was used with the commercial coupling system (Section 2.0). The overall average was 796 N (178 lbs.) with a standard deviation of 85.2 N (19.2 lbs.) for the virgin results. This in itself was a breakthrough because no other known system could survive the Y267 processing. The above compared with the virgin average of 142 N (32.0 lbs.) for the commercial system as shown on Table III for the 288C processing with steel. Because the virgin results of the commercial system were minimal, 142 N, and not much beyond the frictional resistance, there was clear indication that the alternative coupling system should be pursued. With this confirmation the adaptation process to the Y267/steel materials and the geothermal brine environments was initiated.

After chem ageing in synthetic geothermal brine for 166 hrs. at 260C (500F) degradation occurred similar to the commercial system. However, the advantage of the alternate adhesion system was based on the virgin results, knowing that improvements in the thermochemical resistance was needed. It was apparent that good virgin adhesion was a first priority.

TABLE IV. ROD-PULL DATA - SERIES 600 COUPLING SYSTEM

Shop Order	Virgin N (lbs)	Chem Aged N (lbs)
261	707 (159)	
	716 (161)	
	734 (165)	
263		205 (46)
		147 (33)
		80 (18)
273	930 (209)	187 (42)
	814 (183)	405 (90)
	507 (132)	360 (81)
	850 (191)	173 (39)
	885 (199)	423 (95)
	707 (159)	289 (65)
	850 (191)	267 (60)
	890 (200)	400 (90)
	801 (180)	254 (57)
288	801 (180)	
	885 (199)	
	765 (172)	
	734 (165)	
	818 (184)	
	778 (175)	
Mean	796 (178)	266 (59.8)
Standard Deviation	85.2(19.2)	113 (25.3)

Refinements during the adaptation process showed steady progress. After brine aging, early experiments showed that out of the 24 rods only 2 pulled over 178N (40 lbs), the values were 195 and 205N (43.9 and 46.1 lbs.). On a subsequent experiment 17 were over 178N (40 lbs), 10 were over 222N (50 lbs), and 5 were over 356N (80 lbs).

During the adaptation process, inconsistencies in the butt-joint test data became apparent. An experiment was run to investigate new variables which may have caused the inconsistencies but those results were inconclusive with respect to the parameters investigated. The fact that the rod pull data were generally consistent, lead to the suspicion that fabrication procedures of the butt-joint specimens may be a factor.

Analysis of the entire fabrication procedure pointed to the possibility that rubber flow by the adhesion surface was eroding the bonding agent from the butt-joint test surface of the coupon. High shear forces could be generated as the Y267 flows because of its very high viscosity. An experiment designed to investigate that possibility was then performed.

Results confirmed that in-process erosion was at least one of the primary contributors to butt-joint measurement inconsistency. After modifying the way in which the butt-joint mold was loaded with the uncured Y267 EPDM preforms, the data on Table V resulted. In general, the values had very good consistency; in prior inconsistent runs 3.45 MPa (500 psi) was a very good level with many measurements at the 2.07 MPa (300 psi) level.

TABLE V. BUTT JOINT ADHESION RESULTS AFTER MOLDING CHANGE

Y267 EPDM/Steel-Virgin

	System 631	System 644
Individual Values	3.91 MPa (567 psi)	4.57 MPa (662 psi)
	4.38 (635)	3.83 (555)
	4.41 (640)	4.44 (644)
	4.73 (686)	4.67 (677)
	4.41 (640)	4.30 (623)
	4.60 (667)	3.23 (472)
Mean	4.41 MPa (639 psi)	4.13 MPa (599 psi)
Standard Deviation	±0.279 MPa (40.5 psi)	±0.510 MPa (73.9 psi)

Once the coupling system adaptation techniques were narrowed to the most promising ones, and the measurements could be made with reasonably consistency, in depth evaluation of the most promising systems was performed. As shown in Table V, System 631 looked very good with very good consistency, and System 644 was also good.

A final set of two brine ageing tests in L'Garde's standard synthetic geothermal brine (see 2.1.2.) was planned. The first brine aging period was 166 hrs. (about 7 days). The two coupling system alternatives (631 and 644) each with four processing alternatives (1 through 4) were evaluated. The second brine ageing was planned to last 344 hrs (about 14 days) and to evaluate the chosen coupling system, 631, with the same four processing alternatives. It was evaluated with RG 60 steel again, but in addition, with two corrosion resistant alloys, Hastelloy C276 and Proprietary Alloy #1. These were materials of construction to be used for the field tubing prototype.

Table VI summarized those data from the butt-joint specimens run in the above two described autoclave ageing tests. It should be noted that the data associated with the "344 hr" test needs to be considered in a qualified manner because the specimens experienced a burst disk failure after 101 hrs. with an associated "explosive" decompression. It should also be noted that the number of data points measured for each set of conditions is statistically deficient. This was recognized at the time the measurements were being made, however, the number was limited by budget constraints. Because the autoclave was of finite size and a limited number of metal coupons were available (especially the corrosion resistant alloy coupons), conscious consideration was taken to as judiciously as possible select appropriate data points within the allowable number. In some cases virgin conditions were not run and in one case only one proprietary coupon was run because there were no more available. The low number of data points however, were better than was apparent at first glance because correlation existed between the various sets of neighboring data. Thus, if two data points for the same set of conditions were divergent, the neighboring data helped to determine which point was the outlier. It was noted that the high alloy materials generally might not adhere to Y267 EPDM after autoclave ageing as well as steel. This would be expected because they are chemically less reactive. The Proprietary Alloy #1 adhered somewhat better than Hastelloy C276.

TABLE VI. BUTT-JOINT ADHESION MEASUREMENTS

SYSTEM	PROCESS NUMBER							
	1		2		3		4	
	V MPa (psi)	CA MPa (psi)	V MPa (psi)	CA MPa (psi)	V MPa (psi)	CA MPa (psi)	V MPa (psi)	CA MPa (psi)
Steel, RG60								
631 (166 hr)		0.897 (130) 0.779 (113)		0.834 (121) 1.345 (195)		0.537 (77.8) 0.485 (70.3)		0.931 (135) 0.972 (141)
("334" hr)[a]	3.834 (556) 3.669 (532)	0.195 (28.3) b	b 4.862 (705)	0.745 (108) 1.028 (149	6.297 (913) 5.379 (780)	1.179 (171) 0.966 (140)	3.441 (499) 3.497 (507)	1.152 (167) 1.028 (149)
644 (166 hr)		1.055 (153) 0.903 (131)		1.062 (154) 0.807 (117)		0.072 (10.5)		0.503 (73.0) 0.257 (37.2)
Hastelloy C276								
631 ("334" hr)[a]	3.338 (484) 3.759 (545)	b b	4.208 (584) 4.221 (612)	0.296 (42.9) 0.353 (51.2)	4.014 (582) 4.717 (684)	0.253 (36.7) 0.387 (56.1)	4.172 (605) 4.690 (680)	0.369 (53.5) 0.315 (45.7)
Proprietary Alloy #1								
631 ("334" hr)[a]	4.869 (706) 4.400 (638)	0.489 (70.9) 1.441 (209)	3.628 (526) 4.090 (593)	0.497 (72) 0.542 (78.6)		0.438 (63.5)	4.131 (599) 3.572 (578)	0.652 (94.5) 0.603 (87.5)

a = "334" Hr Autoclave ageing terminated due to burst disk failure at 101 hrs. and oven shutdown at 109.5 hrs.

b = Broken during handling

3.3 ADAPTATION CONCLUSIONS

Task 3 was initiated with the knowledge that the commercial coupling system could not survive the Y267 processing, let alone autoclave ageing. With respect to the Series 600 Coupling System, it was known that it was reliable for other applications and conditions, but though it was judged to have a fair chance of meeting the Y267 EPDM Liner requirements, it was not certain it would survive the processing or the brine ageing.

The following are conclusions associated with the adaptation process:

- The Series 600 Coupling System was shown to successfully survive the Y267 EPDM process very well and also to survive brine ageing at 260C (500F) for 166 hrs. with respectable levels of adhesion.

- Direct one-for-one comparison, except they were made and tested at different times, with the commercial coupling system shows over 560% higher rod-pull out force for the virgin case.

- Of the Series 600 options, the specific option System 631/process 4 was selected for the field tubular specimens because of its somewhat better performance after brine ageing. However, the sparsity of the data points does not allow a conclusive determination at this time.

- Coupling system 631/process 3 is perhaps a viable alternative.

- More data points are needed to confirm the precise optimum.

- Further adaptation to produce more consistent adhesion is needed.

4.0 FIELD PROTOTYPE EXPERIMENT (TASK 1)

One of the objectives of the Y267 Liner Program was to fabricate a prototype for testing in an actual geothermal operation. Section 4.0 reports the activities associated with the prototype development and testing.

4.1 FIELD EXPERIMENT

The following items were those which were hoped to be accomplished as a result of the field prototype experiment:

- Demonstrate the overall viability of using Y267 EPDM liner as corrosion protection for steel tubulars in corrosive geothermal brine service.
- Assess Y267 EPDM liner protection of the underlying steel from corrosion.
- Demonstrate that Y267 EPDM can be successfully fabricated into tubing.
- Demonstrate that Y267 EPDM can be durably adhered to the steel.
- Determine the durability of the Y267 EPDM against the brine hydrodynamics.

Consistent with the limited scope of the Program, aspects of the liner problem that were explicitly defined as being outside of the scope of the present Program. These aspects were investigation of lengths beyond the 86 cm (34 in) prototype and interfaces or terminations of the liner at the tubular joints.

4.2 FIELD EXPERIMENT CONSIDERATIONS

This section summarizes some conclusions regarding testing of elastomers for extremely hostile environments. The conclusions were based on several years of testing and developing elastomers in this relatively new 260C (500F) regime for elastomers. Testing elastomers in these very extreme environments is a specialty unto itself.

Given an experimental objective; the complexity, the degree of advance of the state-of-the-art, the degree to which ultimate capability of that which is being tested is being utilized, the extremity of the engineering environments, etc., will place the experiment in some regime within the testing technology level spectrum. If most experiments within the geothermal applied R&D field fall in the 20 to 60 percentile band of the spectrum, generally, experiments dealing with polymeric materials in geothermal environments will fall in a regime trending towards the upper more difficult end of that band. At temperatures of about 230C (450F) and above, the ultimate capacity of these polymeric materials is being approached. They are generally relatively very weak materials compared to metals and at geothermal temperatures the strengths may be on the order of hundreds of psi vs tens of thousands of psi. A testing philosophy of significantly overstressing candidates is good if at least one economic candidate with no significant downside survives. The survivor is then known to have a generous margin of safety defined by the degree of overstressing. However, usually more refined experiments are appropriate in these hostile geothermal regimes, since the luxury of being able to overlook real candidates does not exist because there aren't that many candidates in the first place and those that may survive overstress tests may be very expensive. Consequently, if the experiment too greatly overstresses the polymeric material, failure is likely, and a nonoptimum conclusion is likely. The material that may be more optimum than other alternatives may be eliminated because it was unduly overstressed.

Designing the practical experiment for hostile environments is complex and rightfully deserves due consideration. The most important conclusion to be drawn from the above discussion is that the approach or the philosophy applied to designing the experiment must be influenced by many factors. It would be unwise to operate in the same way for every experiment independent of the unique specifics.

4.3 FIELD TEST PROTOTYPE DESIGN

It may have been desirable to test an entire joint of Y267 lined tubular. The test specimen would have then been long enough such that end effects due to the tubing connections should have been absent from some substantial portion of the length thus providing assessment in an end-effect free zone as well as having the connections where end effects would have been present. For this program this questions was mute, in that catastrophic failure destroyed any data of this nature anyway. However, the program was limited to subscale prototypes.

An objective of the prototype design was to preclude the design effort associated with optimizing operational liner terminations at the couplings. This was accomplished by simply terminating against a corrosion resistant alloy. The test section was fabricated in a 86.4 cm (34 inch) length, allowing cure and postcure to occur in an existing oven. Larger parts would have had to be cured in a new larger or outside oven increasing the costs significantly, but worse, hindering the flexibility and the ability to modify, ponder, and perfect the manufacturing process that an inhouse oven provides. The pressure to get in and out of an outside facility, coordinating with another organization, synchronizing with their availability, and the expense of travel and shipment can be extremely limiting to a development effort. An associated limitation of this approach was a temperature limit of 260C (500F) which precluded postcuring at the standard 288C (550F) temperature.

Figure 5 (drawing 11056) shows a sketch of the field prototype designed and built by Unocal except for the Y267 liner. The basic envelope was defined by L'Garde and the detail design performed by Unocal. It simulates an 8-5/8 inch 36 ppf tubular that is 87.4 cm (34.41 in) long. The tubular is lined with 61.8 cm (24.33 in) of Y267 EPDM with 0.635 cm (.25 in) wall thickness. The wall thickness was based on a judgement considering the requirements for impermeability, processing and material costs. Future optimization of the ultimate thickness is required. All metal parts are corrosion resistant alloys except the mild steel corrosion witness cylinder placed behind the Y267 EPDM to simulate the conditions that a steel tubular would experience. The steel cylinder was designed to be electrically insulated from the tubular with Teflon (PTFE) and the Y267 O-rings to prevent formation of galvanic coupling between the dissimilar metals, and subsequent galvanic corrosion. However, the Y267 EPDM is somewhat electrically conductive so perfect insulation was not achieved. Because the Y267 EPDM was not perfectly adhered to the tubular, the 18.6 cm (7.325 in) ID ring was added to pinch the end of the Y267 cylinder outward to seal and prevent leakage of the brine behind the Y267 EPDM. Any brine behind the Y267 EPDM other than that which may permeate through it would invalidate the experiment.

This field test prototype was then welded to a tubular extension or stinger which mounted to a flanged coupling in the host brine line. This was accomplished by welding a plate to the end of the stinger which was sandwiched between the two flange faces at the tubing joint. Thus, the prototype test section was mounted concentrically within the existing tubing and full line flow went through it.

4.4 Y267 LINER FIELD PROTOTYPE FABRICATION

This subsection reports all the activity associated with preparing and fabricating the Y267 lined field test prototype.

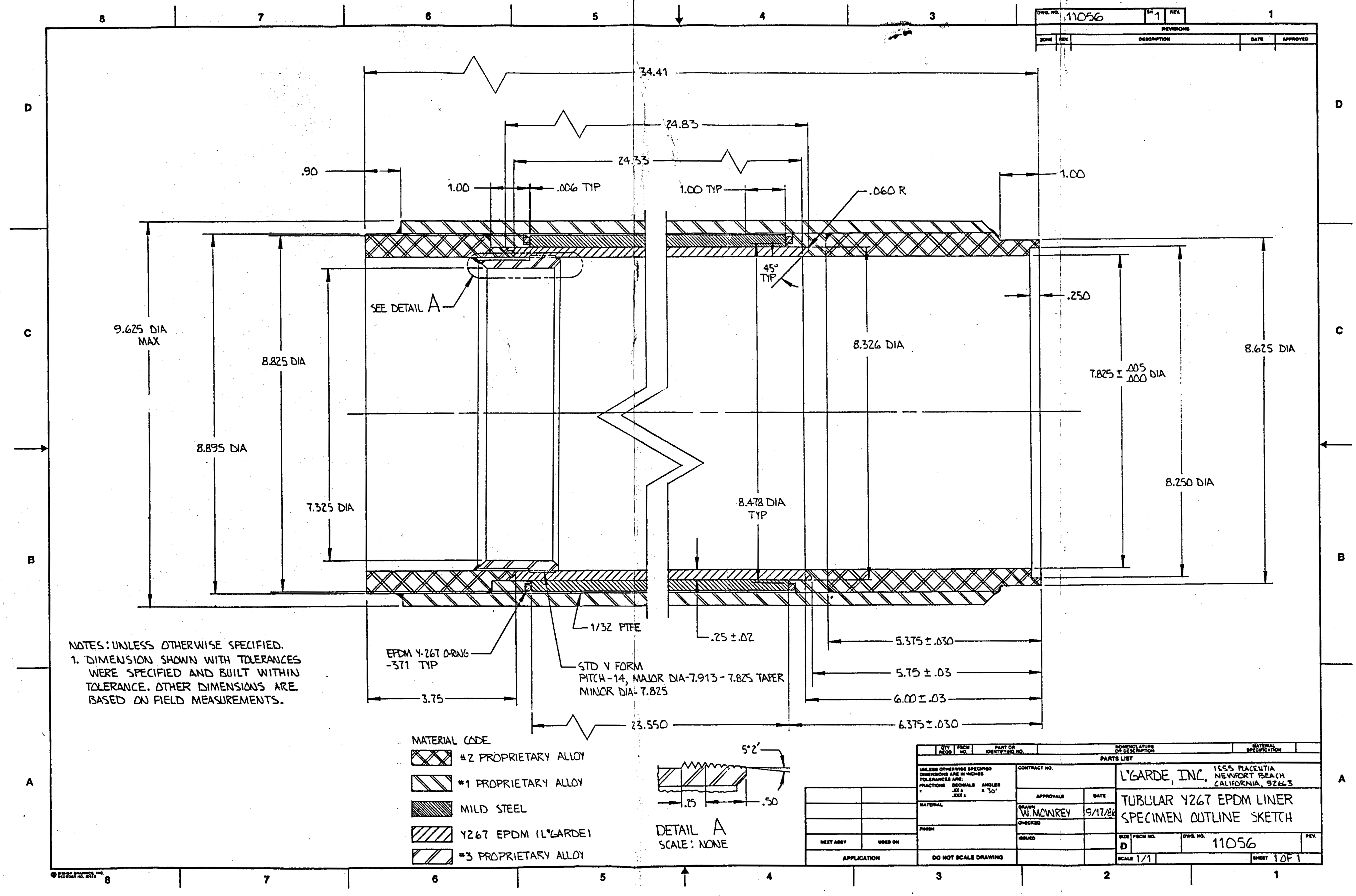


Figure 5. Tubular Y267 EPDM Liner Specimen Outline Sketch

4.4.1 Prototype Approach

A streamlined fabrication effort was planned assuming no scale-up development of the coupling system was required in going from the laboratory coupon work to the full scale fabrication of a prototype. The smooth scale-up transition did not occur. The actual experience is described in following subsections.

The original plan called for delivery of two identical prototypes for testing in actual geothermal operational field conditions. Preparatory to fabricating the two deliverables, two practice moldings were planned to work out the details of molding the liners into actual 8-5/8 inch 36 ppf steel tubing.

The responsibilities for the fabrication were divided between L'Garde and Unocal with Unocal preparing and providing the tubular field specimen which included installation of the corrosion witness cylinder, and with L'Garde doing the balance which included preparing the Y267 EPDM, elastomer, and molding the Y267 into the prototype.

A major concern associated with the molding existed from the outstart. Molding material onto the ID of tubing is usually a relatively complex procedure that is particularly difficult when the material is viscous, the wall thickness thin, and the ID is small. Dynamic and static means are possible using centrifugal force or a mechanical means to apply the molding force. For the Y267 Liner Program, mechanical means were used to provide the molding force because it was the only realistic consideration within the scope of the effort. Mechanical means pose some difficulties because the tooling must have a reduced diameter to allow insertion into the tubing and then it must expand to provide the molding pressure. The expansion can be accomplished mechanically or through inflation. Both techniques are possible but become geometrically more difficult as diameter decreases - at some point it becomes impossible to fabricate tooling that can be inserted into the space available and then expanded to the necessary diameter. The relatively long 61.8 cm (24.33in) and thin .635 cm (.25in) dimensions of the liner, made this task difficult.

An early judgment was made to use mechanical as opposed to inflatable tooling, because more developmental problems were anticipated with inflatable tooling which could not be tolerated with the limited scope. This weighed against the probability that in production inflatable tooling would probably be used. Thus, mechanical compression molding tooling was designed which conceptually consists of "barrel stave-like" elements which are forced outward by an internal conical wedging pin. The degree of expansion allowable was limited, because the greater the percentage expansion the larger the resultant gaps between the "barrel staves". The other real concern was due to the shallow cone angle of the wedging pin, which presented the significant probability of irretrievably jamming the pin and in turn the tooling inside the tubing.

4.4.2 Fabrication

The fabrication plan included working out the molding techniques with two trials, then molding the deliverable field test prototypes.

A. Practice Moldings

Full scale practice moldings of Y267 EPDM into practice mild steel tubing using actual tooling and without the coupling system were performed to perfect the molding techniques. The Y267 EPDM was calendered into three thicknesses within the range of 2.92 to 3.68 mm (0.115 to 0.145 inches) to provide a choice of several combinations to provide the precise amount of elastomer required for a good molding.

The first practice molding was approached with anxiety and trepidation because of the real probability that the tooling may be irretrievably stuck in the tubing. Though the tooling was somewhat difficult to remove, especially the first time, it was successfully removed every time.

The first practice molding turned out very well, except that insufficient rubber was loaded into the mold. There was not sufficient rubber to fill out the ends of the Y267 section and its longitudinal joints where the flat calendered sheets butt together.

The second practice molding resulted in a practice prototype that indicated the molding process was feasible and successful. The result is shown in Figure 1. No blemishes appeared on the surface, there was no evidence of the longitudinal splice where the sheet preforms were required to join and knit, and a perfect chamfer was formed at the end that mates to a metal ring. It also appeared that the stiffness of the Y267 EPDM compound in both its cured and uncured states helped in processing. The stiffness tends to help hold the preform in place during set-up, and after the part is completed, it provides mechanical strength which holds the Y267 EPDM in place against the ID of the pipe.

Once success was achieved with the second practice molding it was decided to proceed further and to postcure the Y267 EPDM Liner. No coupling agent was used to bond the Y267 EPDM to the steel on the first two practice moldings, since their purpose was to work out the molding process. During postcure of the Y267 EPDM, the elastomeric part is usually removed from the mold and fully exposed to the postcure atmosphere. This allows the lower molecular weight components to be more freely extracted from the elastomer. Similar to the smaller molded parts, the tooling was removed from the tubing to give a maximum exposure of the elastomer to the postcure environment. During the postcure as the volatiles were formed, they pressurized the uncoupled interface between the steel tubing and the elastomer, and formed two distinct bubbles.

It was anticipated that bubbles would not occur when the actual deliverable parts were to be made because the coupling system would keep the elastomer adhered to the tubing. In addition, for future parts the plan was to step up the postcure temperature more slowly to reduce the rate of generation of volatiles and, thus, the resulting interfacial pressures. Furthermore, if a problem persisted with the coupling system in place, a fall-back position of leaving the tooling in place during the postcure could be used. The bubbling was the first indication of problems to come.

Because of the above uncertainty, it was concluded that it would be prudent to try another practice molding but with the coupling system to indicate whether a bubbling/separation problem existed with the coupling system in place. For this practice and the deliverable prototypes, the System 631/Process 4 was incorporated (see 3.3). In that only two practice moldings were budgeted, it was decided to reduce the deliverable field prototypes to one, to allow for the additional practice molding. Furthermore, it was decided to perform a double postcure on it. The first with the tooling in place to confine the rubber while it coupled to the metal, and the second with the tooling removed. With the one prototype molding two questions were planned to be answered; that a part could be made bubble free with the tooling in place, and that it would remain bubble free through the second postcure once the tooling was removed thus allowing the low molecular components in the Y267 EPDM to be more freely extracted from an exposed surface.

The first postcure was clouded by the fact that a 0.3175 cm (0.125 in) hole was drilled through the wall of the tubing at the center of the length for this and previous parts. A thermocouple was typically inserted through this hole to monitor the temperature of the rubber and no problems occurred on previous tries. However, on this run rubber was flowed out the hole carrying the thermocouple with it. The reason for the difference on this run could not be determined, however, it is possible that this occurred because friction between the Y267 EPDM and the tubing was reduced by the coupling system which was not present on previous runs, allowing freer flow of the rubber. During the processing 156 grams of rubber flowed through this hole.

The first post cure with tooling in place resulted in very minor bubbling at the extreme end of the part where the molding pressure on the rubber was the lowest. The bubbling was barely discernible, but confirmed acoustically by tapping the liner. It was postulated that the rubber escaping out the hole prevented sufficient pressure from feeding down to the extremity of the part.

The second post cure without tooling occurred pretty much as anticipated. The barely discernible bubble grew to about twice the area which was still a minor fraction of the total area and the bubble became definitely discernible. It was very gratifying to learn that other than the growth of the original bubble, no new bubbling occurred. However, a three mm (one-eighth inch) break in the rubber was observed at the bubble where trapped volatiles escaped. The break was attributed to weak planes of insufficient crosslinking which probably occurred because of insufficient molding pressure. This same phenomenon was experienced earlier on thick packer elements molded for a major service company.

It was not certain why there was an adhesion failure where the bubbling occured. It was postulated that the coupling system might have flowed to a low spot during preheating of the metal parts, but subsequent tests on coupons rendered this improbable. On deliverable parts there would not be a thermocouple hole and at that point in time L'Garde expected that the bubbling problem would be cured once the elastomer was fully trapped during molding.

Destructive examination of the practice molding was performed to evaluate the adhesion. It consisted of cutting the liner into longitudinal strips and peeling the strips off with a force directed along the adhered portion. The liner was split into quarters with a one inch wide strip peeled approximately every 90 degrees.

At first, peel tests were made without quantitative measure because, based on the observations after the first postcure, it was expected that the adhesion would be inconsequential. However, it was noted that there was a significant level of adhesion, though not up to the coupon developed level, that was greater than the negligible adhesion observed on the one partial strip (about 2.54 x 25.4 cm (1 x 10 in) pulled after the first postcure. Typically, the strips broke before being completely removed. There was also some limited evidence of cohesive failure at the bondline, i.e., Y267 EPDM stuck to and remained on the steel. Subsequently, each of the larger four segments (about 14 cm (5.5 in) wide) were peeled with one person maintaining his maximum peeling force while a second pried the Y267

EPDM free with a screwdriver. In all cases, peeling only occurred in the local vicinity of the screwdriver and sustained peels could not be achieved. The adhesion appeared best away from the vicinity of the strip pulled after the first postcure.

With the above evidence of some significant levels of adhesion, quantitative measurements were then undertaken. 2.54 cm (1 in) strips were cut from the remaining portions of the 14 cm (5.5 in) larger segments. Three 2.54 cm (1 in) strips were measured, two from the segment which qualitatively appeared to have the poorest adhesion, and one from the segment which appeared to have the best as shown in Table VII.

TABLE VII. QUANTITATIVE ADHESION TESTS ON THE LABORATORY PRACTICE PROTOTYPE FOLLOWING SECOND POST-CURE

Segment	Peel Force, N/cm (lbs/in)	Comment
#8 (#7 side)	70 (40)	Adhesion previously appeared worst
#8 (#1 side)	63 (36)	Adhesion previously appeared worst
#4	17.5 (10)	Adhesion previously appeared best

There is an obvious discrepancy in the Table VII data which cannot be explained at this time, what originally appeared worse measured better. However, these quantitative measurements show peel forces per unit width of the bond that were significant at the 17.5 N/cm (10 lb/in) level and very respectable at the 70 N/cm (40 lb/in) range.

In addition to the above diagnostics, the effect of the prolonged cure, use of different calender batch of Y267, and the probability of the coupling agent flowing away during preheating of the metal parts were investigated. The above parameters were the most probable differences between the coupon tests and the practice prototype that were identified. Butt joint coupons were fabricated with Y267 from both calendered batches (both from the same Y267 mix) and the cure and postcure were stepped over a prolonged period of time at prototype temperatures to simulate the cycle the practice prototype experienced. In addition, part of the coupons were preheated before being placed into the mold to also simulate the practice conditions. An experiment showed absolutely no flow of the coupling agent while undergoing the preheat cycle. The results of the coupon tests are shown in Table VIII and the prolonged postcure cycle in Table IX.

TABLE VIII. PROLONGED POST-CURE BUTT JOINT ADHESION TESTS

Calendered Batch mm (in)	Butt Joint Adhesion Preheat[a] MPa (psi)	No Preheat MPa (psi)
2.92 (.115) thk	2.70 (392)	2.85 (413)
	2.89 (419)	4.12 (598)
		4.63 (671)
Mean	2.80 (406)	3.87 (561)
3.68 (.145) thk	2.66 (386)	4.70 (682)
	2.58 (374)	3.43 (497)
		3.14 (452)
Mean	2.62 (380)	3.75 (544)
	Overall Mean	3.81 (552)

[a]2 hrs. @ 52C (125F)

TABLE IX. PROLONGED POSTCURE

Temperature, C (F)	Time, hrs.
149 (300)	2
177 (350)	2
204 (400)	2
232 (450)	2
260 (500)	5

The conclusions of the above coupon tests are as follows:

- Preheat appears to degrade the adhesion somewhat. Coupons were consistently at the 2.76 MPa (400 psi) strength level vs the 3.87-3.75 MPa (561-544 psi) means.
- There appears to be no significant difference between the two calendered batches, means were 3.87 and 3.75 MPa (561 and 544 psi).
- There appears to be no significant effect due to the long cure and postcure cycles. The mean of the 6 data points without preheat was 3.81 MPa (552 psi). There were two datapoints somewhat below the 3.45 MPa (500 psi) level at 2.85 and 3.14 MPa (413 and 452 psi).

The overall conclusions made after completing the practice prototype with coupling were the following:

- Adhesion was not sufficiently reliable for operational purposes and the process needs further refinement. However, the adhesion was significantly better than anticipated based on earlier qualitative measurements, and was judged to be sufficient for a field test prototype.

- There was apparent improvement in the adhesion as a result of the second postcure demonstrated by the Table VII results.

- Absence of the thermocouple access hole in the field test prototype was anticipated to solve problems with bubbling and weak planes in the Y267 experienced on the practice prototype.

B. Field Test Prototype Fabrication

The fabrication of the field prototype was shared between Unocal and L'Garde. Unocal fabricated the high alloy tubular with the mild steel corrosion witness cylinder. L'Garde prepared the part for the coupling system, applied the coupling system, and molded the Y267 liner into place. Unocal then installed a closure ring against the end of the molded Y267 EPDM liner and an extension tubing to provide for the concentric mounting within an existing brine supply line.

The following were the steps taken to fabricate the Y267 Liner into the field test prototype:

1. Clean the bonding surfaces of tubing, surfaces were newly machined by Unocal.

2. Sand bonding surfaces with 180-220 grit sandpaper.

3. Clean surfaces again.

4. Apply basecoat to prepare the metal surfaces.

5. Dry overnight in 107C (225F) oven with N_2 purge.

6. Apply coupling system on day prior to molding in Y267 EPDM.

7. Cure Y267 EPDM for 60 min. at 177C (350F)

8. Start N_2 purge and proceed directly into #1 postcure with tooling still in place according to the following schedule:

   177C (350F) 2h 20m

   204C (400F) 2h

   232C (450F) 2h

   260C (500F) 5h (over maximum)

9. Cool, remove tooling and inspect.

The above nine steps occurred routinely except there was some difficulty removing the tooling. The tooling had been chrome plated and then coated with teflon but on the last practice prototype the coating was destroyed when the tooling was left in during the first postcure. Apparently the combination of the molding force on the teflon plus the 260C (500F) temperature caused the teflon to decompose and form highly corrosive acid which lifted the chrome plating and left the steel exposed. No problem occurred with previous practice prototypes at the molding temperature 177C (350F). L'Garde experienced this phenomenon once before but at 371C (700F), close to the sintering temperature of TFE teflon. The corroded surface of the steel tooling was then protected with the Series 600 Coupling System basecoat and was released with silicone release agent. Even with this surface preparation, there was sufficient adhesion between the Y267 and the released tooling which caused some difficulty in removing the tooling.

Once the tooling was removed it was apparent that areas of Y267/Steel nonadhesion existed. Separation bubbles, though not prominent, could be discerned. They were slightly higher and over substantially more area then on the practice prototype with coupling. The separations were confirmed acoustically by tapping the Y267 EPDM surface. Figure 6 shows the tubular section which contains the Y267 Liner. The crosshatched area marks the region of nonadhesion. Approximately 50-70% of the lined area was not adhered. The 60.96 cm (24 in) long rubber section leaves about 12.7 cm (5 in) at either end of the 87.4 cm (34.41 in) tubing unlined. Note that the end on the floor was adhered around the total circumference.

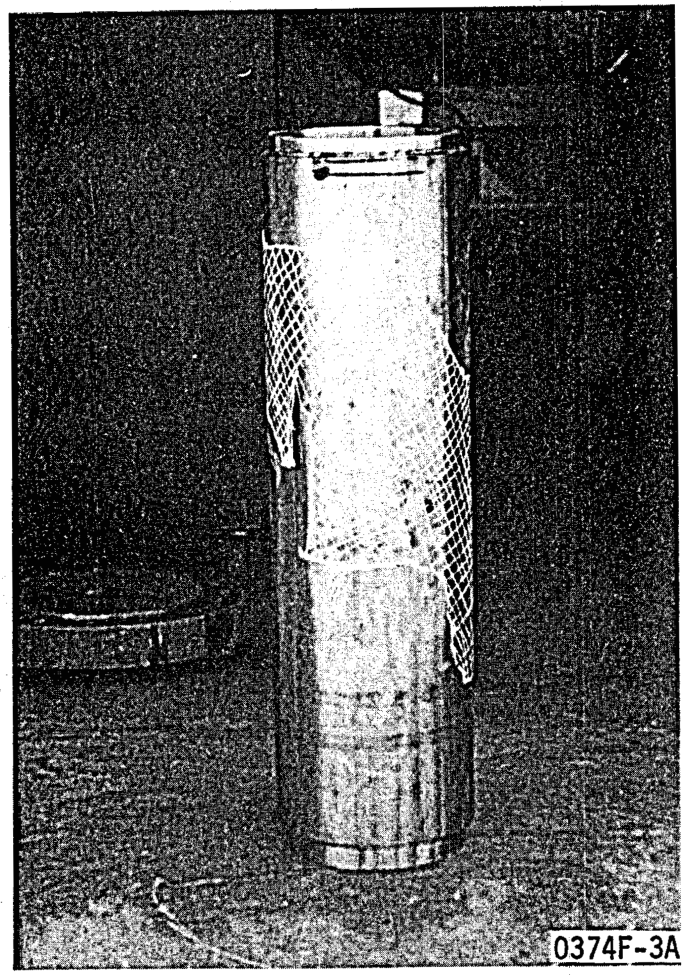


Figure 6. Field Prototype Showing Nonadhesion Identified During Fabrication

With the nonadhesion that was apparent after the first postcure, it was obvious that the scale-up processing problems had not been circumvented. A significant difference between the coupons and the tubing is the orientation of the adhesion surfaces with respect to the gravity vector while the coupling system is being applied and dried. The coupons were typically dried with the adhesion surface approximately parallel to the ground, and the tubing was dried horizontally. Consequently, the tubing walls had the total spectrum of orientation from vertical to horizontal face up and down versus face down with the coupons. This aspect of the scale-up needs future developmental attention.

Given the problem with the nonadhesion after the first postcure, the question emerged whether the planned second postcure should be carried out. It was known with a fair degree of confidence that under the circumstances the second postcure with the tooling removed would result in bubbling of the liner and thus render it unusable for field test purposes.

It was decided to forgo the second postcure, install an internal ring at the end of the Y267 EPDM Liner where nonadhesion was apparent to mechanically hold the elastomer in place and deploy the prototype in a field test. It was reasoned that this expediency might provide some data versus the alternative of having no field data whatsoever. Details of the internal ring which was installed to squeeze the Y267 EPDM against the ID of the tubing are described in the design paragraph 4.3.

Since the second postcure was omitted there was some question as to the effect on the Y267 properties. Based on previous experience with Y267 EPDM, it was felt with a fair degree of confidence that a partially or nonpostcured part placed into a geothermal environment would tend to postcure in situ. The primary difference would be the extent of the extraction of low molecular weight molecules because of the presence of brine under pressure. Since this was essentially the circumstance presented, an experiment was run to investigate this phenomenon. Both postcured (standard Y267 procedure) and non postcured Y267 EPDM were aged in synthetic geothermal brine in an autoclave at 260C (500F) for 118 hours to determine if a significant difference in properties resulted. The results of the experiment are summarized in Table X. The reason the data are shown is strictly to demonstrate that catastrophic degradation of properties did not occur with in situ postcure. The statistical summary data are shown to provide a summary means of comparison and no deeper interpretation should be made especially since there are only three data points per case.

TABLE X. IN SITU BRINE POSTCURE AUTOCLAVE EXPERIMENT AT 260C

	Y267 STD PROCESS			Y267 - NONPOSTCURED	
	Ult. Tensile MPa (psi)	Ult. Elong. %		Ult. Tensile MPa (psi)	Ult. Elong. %
	16.14 (2341)	122		15.22 (2207)	137
	15.99 (2319)	127		20.01 (2902)	170
	15.43 (2238)	122		16.97 (2460)	147
Mean	15.86 (2299)	123.7	Mean	17.4 (2523)	151.3
Std. Dev.	±.37 (54.2)	±2.9	Std. Dev.	±2.43 (352)	±16.9

The experiment indicates that in situ postcuring should not result in substantially different properties. In fact higher strengths are indicated, though the properties are less uniform than with the standard Y267 postcure. In any event, the strength requirements for a liner are extremely minimal and only a catastrophic degradation of properties, which did not occur, would be a concern.

4.5 FIELD EXPERIMENT RESULTS

The objective of the field experiment was to deploy a representative prototype, to have it perform for an extended period of time, and to recover it in such a condition that it demonstrated the viability of Y267 EPDM lined tubing with very little doubt. The results did not meet the ideal objectives, however, important facts were learned from this experiment.

4.5.1 Test Conditions

The Y267 EPDM field prototype assembly was inserted into a brine supply line from a single Unocal well at their Salton Sea facility. The entire flow coming from the designated well passed through the liner prototype. The geothermal brine was typically at about 246C (475F) with an average total dissolved solids content of 233,000 ppm.

Figure 7 shows the duty cycle experienced by the liner. About 6 days elapsed during which flow was established 3 times for 1.25 to 2.5 hrs. before relative steady state was established. This was maintained for about 7 days. After 7 days the mass rate was throttled back about 25% to about 75% for purposes independent of the Y267 EPDM liner experiment. It then ran about 4 days at the reduced rate after which the flow was stopped. Then the flow was re-established 4 times for 0.5 to 1.0 hrs.

None of the changes in the conditions experienced were driven by the design part of the liner experiment, all were for other reasons. Ideally, from the standpoint of the liner evaluation the desirable duty cycle would have been a relative steady state flow at full mass rate for a period of several months.

4.5.2 Results & Observations

This paragraph describes the observations, measurements and questions raised after recovery of the liner prototype.

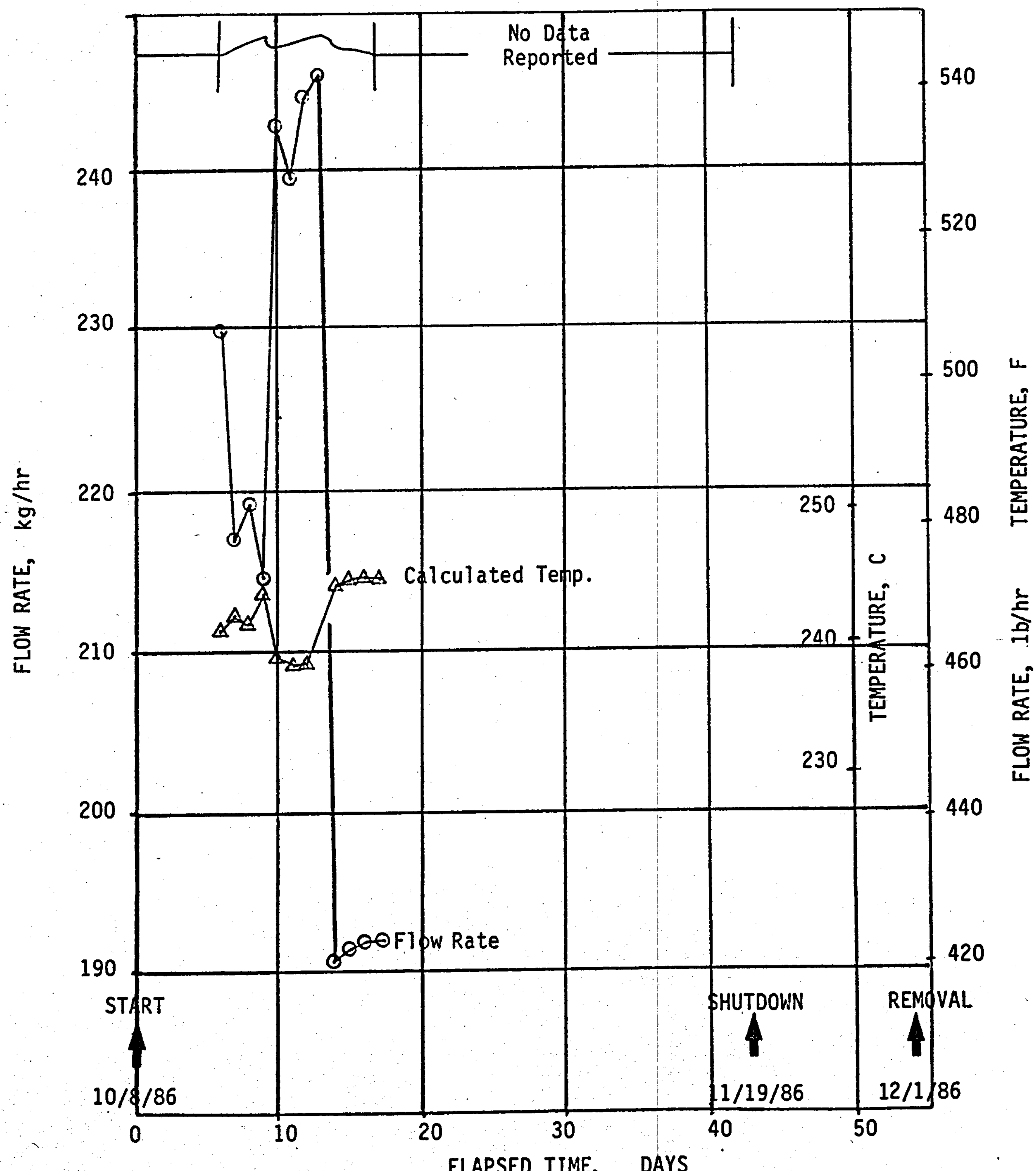


Figure 7. Field Test Prototype Duty Cycle

A. Post Mortem Observations

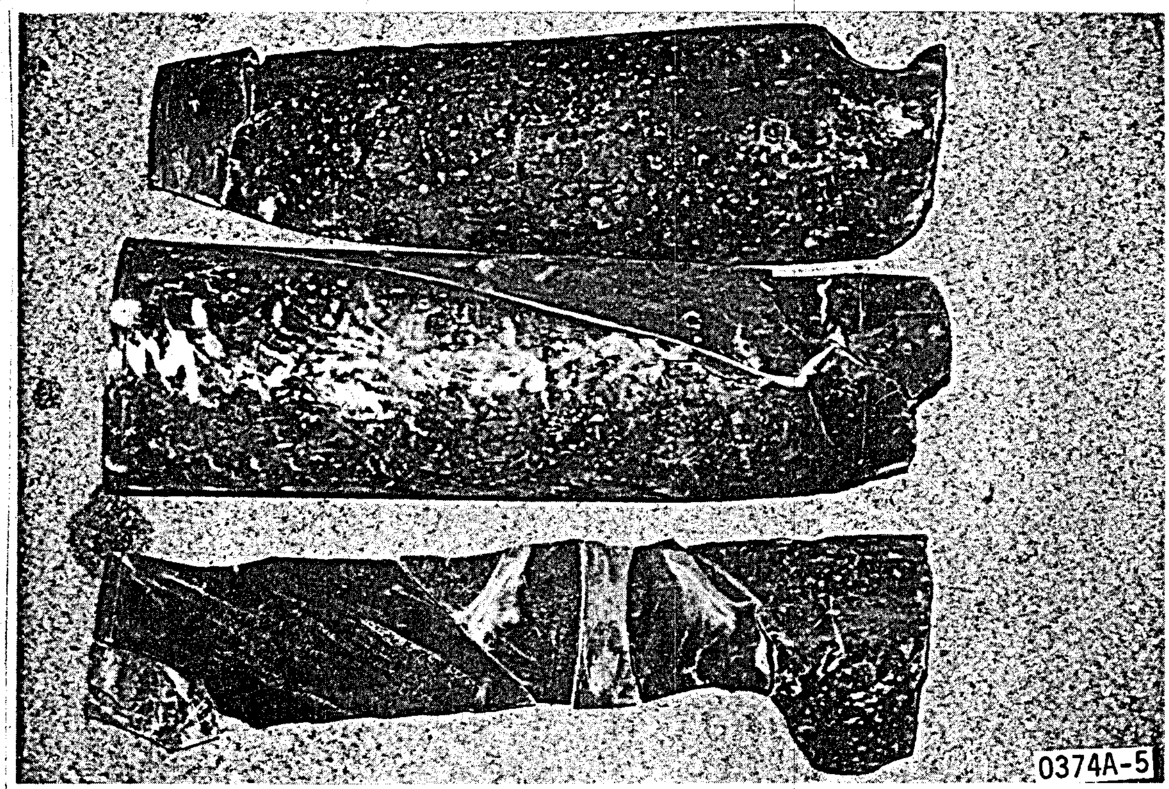
When the prototype test section was removed from the brine supply line it was noted that virtually all of the Y267 EPDM liner was gone. Some small amount of Y267 remained pinched behind the retaining ring and when removed bright metal showing no signs of fluid penetration or corrosion was revealed. The liner which was recovered from an in-line strainer just downstream of the test section was torn into about 10 parts Virtually the entire Y267 EPDM liner was recovered.

Figure 8 shows two views of those parts; the inside and outside diameters. There were two major longitudinal strips; one about 180 degrees of the circumference and the second about 90 degrees. In addition to the two major pieces there were about eight smaller pieces. The ID surfaces were relatively uniformly black, while the OD surfaces showed interesting patterns apparently created by chemical deposits.

The largest piece shows a diagonal, relatively straight deposition line along the OD. Examination suggested the piece adhered to the tubing for sometime on the black side of the diagonal while the Y267 EPDM on the opposite side of the diagonal was separated. The white diagonal line was created by the apparent deposition of chemicals suggesting a crevice was created at the interfaces between the adhered and nonadhered Y267 EPDM. Two cracks through the thickness parallel to the diagonal line at the broken end indicating that the Y267 EPDM was folded back on itself with a significant force probably while the black surface was still adhered. The ID side of this same part has a cracked pattern in the thin varnish-like surface coating similar in appearance to parched land.

There is nothing in the run data in paragraph 4.5.1 which provides any hint regarding when the liner was torn from the tubing it lined. Pressure drops across the strainer were not available and the general decrease in mass rate on 22 October 1986 is attributable to an operating change. The other mass rate variations are too uncertain to provide any semblance of a reliable indicator.

During the short run period there were several increases and decreases in the flow rate not represented in the data. It was not known what effect the excessive cycling had on the prototype, but it was especially vulnerable because the elastomeric liner was not completely bonded to the steel. It was highly probable that there were differential pressure reversals across the liner when the line was shut down, or adjusted. Any reductions in line pressure after a sustained run could



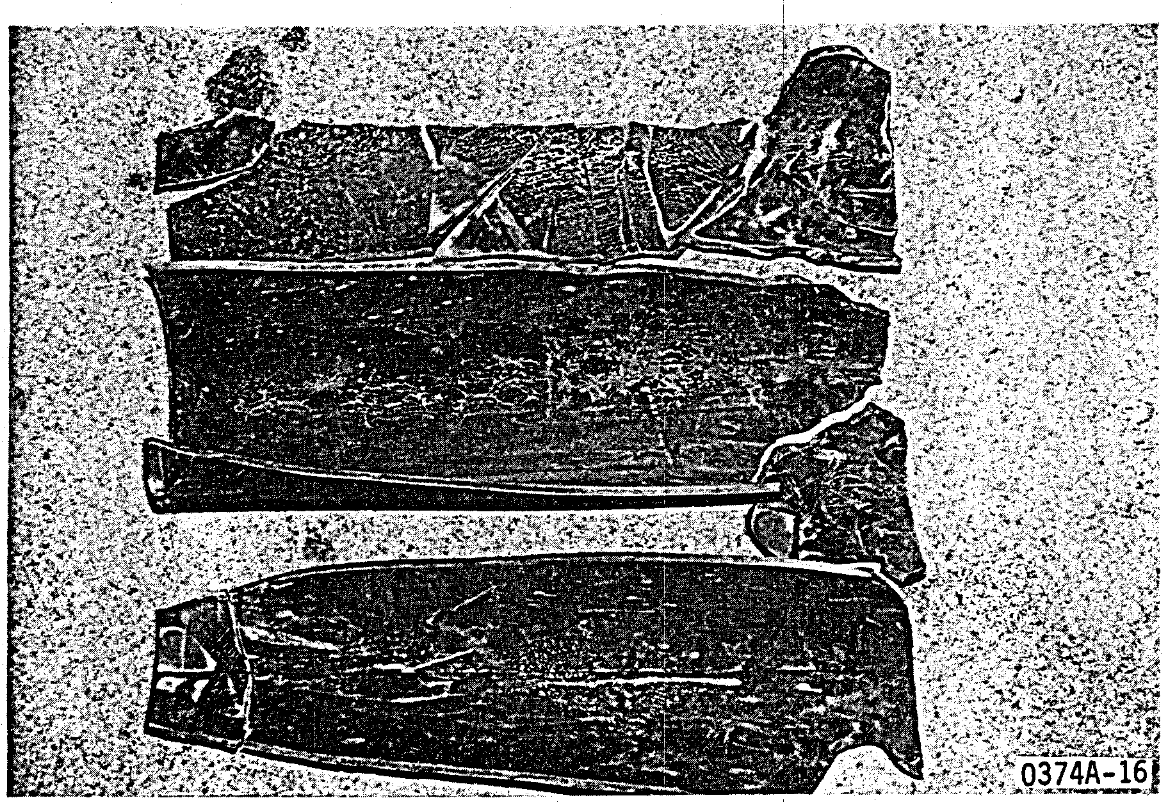


Figure 8. Two Views of Y267 Liner Pieces Recovered From Salton Sea Test

cause any gas trapped behind the liner to balloon it towards the center of the tubing as the pressure reduces.

In addition to observations associated with the Y267 EPDM Liner, there were interesting observations associated with the steel lined tubing. There was a distinct corrosion pattern on the steel which correlates with the pattern of nonadhesion marked onto the tubing prior to the field experiment. Figure 9 shows that correlation between the nonadhesion pattern shown as marked on the taller main tubing and the corrosion pattern experienced and marked on the shorter steel liner.

Figure 9. Similarity of Nonadhesion Pattern (Left) and Low Corrosion Pattern (Right)

There appeared to be a direct correlation between the areas of nonadhesion and of low corrosion. Apparently, when adhesion was achieved with the Y267 EPDM and then the bond was broken, the failure occurred at the steel, thus exposing the steel to the brine. When adhesion was never achieved with the Y267 EPDM apparently the coupling system remained intact and adhered to the steel, thus protecting the steel from corrosion. Hence, the coupling system itself seemed to afford some level of corrosive protection which may have been significant.

4.5.3 Y267 EPDM Measured Data

With prototypes there was limited opportunity to obtain good post test measurements. Two types of measurements, tensile and hardness, were obtained on the Y267 EPDM, liner.

A. Tensile Measurements

Tensile measurements were made on the recovered Y267 EPDM liner. Ten ASTM Die C dogbones were cut from the largest fragment which had the diagonal deposition line described in paragraph 4.5.2. The specimens were cut in the circumferential direction which was the direction of feed through the calender during the sheet fabrication processing of the Y267 stock. Thus, the tensile strength in the calendering direction was measured. Figure 10 shows the general location of the ten tensile specimens.

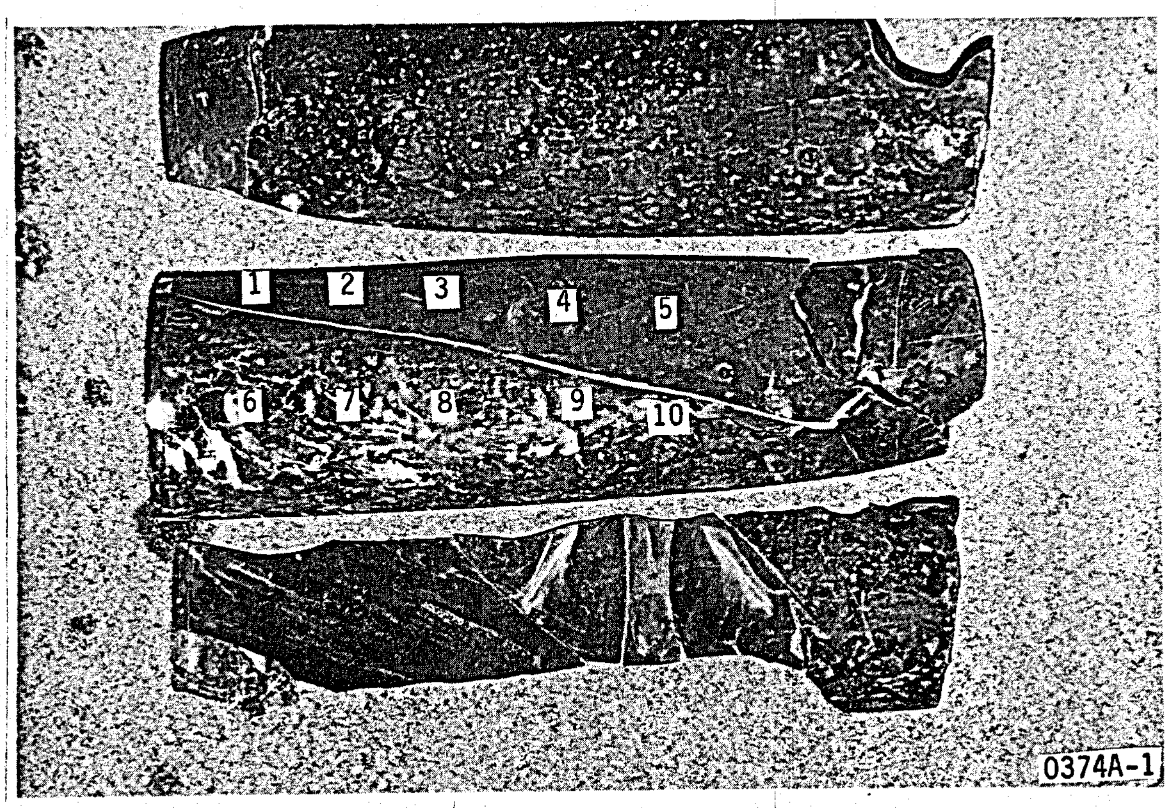


Figure 10. Location of Ten Tensile Specimens on Retrieved Liner are Shown

A set of five measurements on either side of the diagonal were taken to determine if properties differed from one side to the other in that it was postulated they experienced different exposure. Table XI shows the results of these tensile measurements with elongations measured with extensometers.

Note that the specimens were cut from material which sustained forces severe enough to crack the material through its thickness. Consequently, a broad scatter in the data was expected. In addition, there were two anomalous points, specimens 2 and 3. Inspection of 2 and 3 showed that there was not a clean break through the thickness of the specimen, rather each side broke at a different point with a longitudinal break plane connecting the two surface breaks. Hence, these two points were statistical outliers and were not considered to be valid data points. Apparently there was a flaw in the surface on one side of the specimen causing a premature break through part of the thickness.

Considering specimens 1, 4, and 5 to be representative of one side of the diagonal, and specimens 6 through 10 the other side, a comparative analysis was performed. The mean ultimate tensile strengths as shown in Table XI are 8.43 vs 8.12 MPa with respective standard deviations of 1.39 and 0.85 MPa. In like manner the mean ultimate elongations are 122% vs 114% with respective standard deviations of 21.4% and 10.9%. These values were

TABLE XI. Y267 LINER POST MORTEM TENSILE MEASUREMENTS

Specimen	Ult. Tensile Str. MPa (psi)	Ult. Elongation, %
1	7.13 (1034)	103
~~2~~	~~4.72 (685)~~	~~32~~
~~3~~	~~4.56 (661)~~	~~35~~
4	9.89 (1434)	145
5	8.26 (1197)	117
	Mean = 8.43 (1222)	Mean = 122
	Std. Dev. = ±1.39 (201)	Std. Dev. = ±21.4
6	9.39 (1362)	128
7	7.21 (1045)	100
8	8.21 (1190)	115
9	8.31 (1205)	121
10	7.50 (1087)	108
	Mean = 8.12 (1178)	Mean x = 114
	Std. Dev. = ±.85 (123)	Std. Dev. = ±10.9

reasonably close considering normal measurement accuracy, which indicated there was negligible difference in the elastomer on each side of the diagonal deposition line.

Unfortunately these tensile measurements were not comparable to virgin Y267 EPDM so a direct comparison of the field aged Y267 could not be made to the known virgin properties of Y267 EPDM which included a minimum ultimate tensile strength of 13.1 MPa (1700psi) and elongation of 105% using extensometers. There were two significant differences. First, the Y267 liner could not be postcured according to the Y267 specification. Because of the size of the prototype and economic constraints the Y267 liner was postcured at 260C (500F) as opposed to the standard 288C (550F). In addition, because of nonadhesion bubbling problem, the intended second postcure cycle with the tooling removed was not carried out as discussed in 2.4.2B. The effect of less postcure was that full strength and hardness of the compound was not achieved. The second significant difference was that the tensile specimens were cut from parent material which is 0.66-0.686 cm (0.260-0.270 in.) thick compared to the standard ASTM specimen thickness of 0.195 cm (0.077 in). The third significant difference was that the rubber was physically abused by the flow hydrodynamics especially as it was being torn from the prototype.

In any event, the resulting tensile properties after field exposure shown above without the outliers indicate that the Y267 EPDM survived the environment well, even with the apparently mechanically violent experience it went through. Past experience shows that the Y267 EPDM is relatively unaffected by brine at the temperatures experienced in this test. Consequently, the best assumption at this point was that the resulting tensile properties which were very respectable, but somewhat below Y267 EPDM standard minimum virgin properties of about 11.72 MPa (1700 psi) was a result of incomplete postcure and the violent mechanical experience. However, even the lower properties should be sufficient for a liner because when it is operating properly it experiences minimal stress.

B. Shore A Hardness Measurements

Since the tensile measurements are destructive tests, it was not possible to measure the tensile properties of the field test liner prototype prior to being field tested. On the other hand, because hardness testing is nondestructive, it was possible to obtain those measurements before and after field exposure.

Just prior to the delivery of the field prototype to Unocal, Shore A hardness measurements were made. Measurements were made along four longitudes at each 90 degree interval. Measurements could only be made on the ID surface of the elastomer as shown in Figure 11. Sixteen measurements were made, four on each longitude, which resulted in a mean Shore A value of 65.9 with a standard deviation of ± 3.28 and a range from 60 to 70. The hardness is significantly below the usual 90 for standard Y267 EPDM, but this was expected because a significant fraction of the final strength and hardness of the Y267 EPDM develops during the postcure and the prototype was not fully postcured.

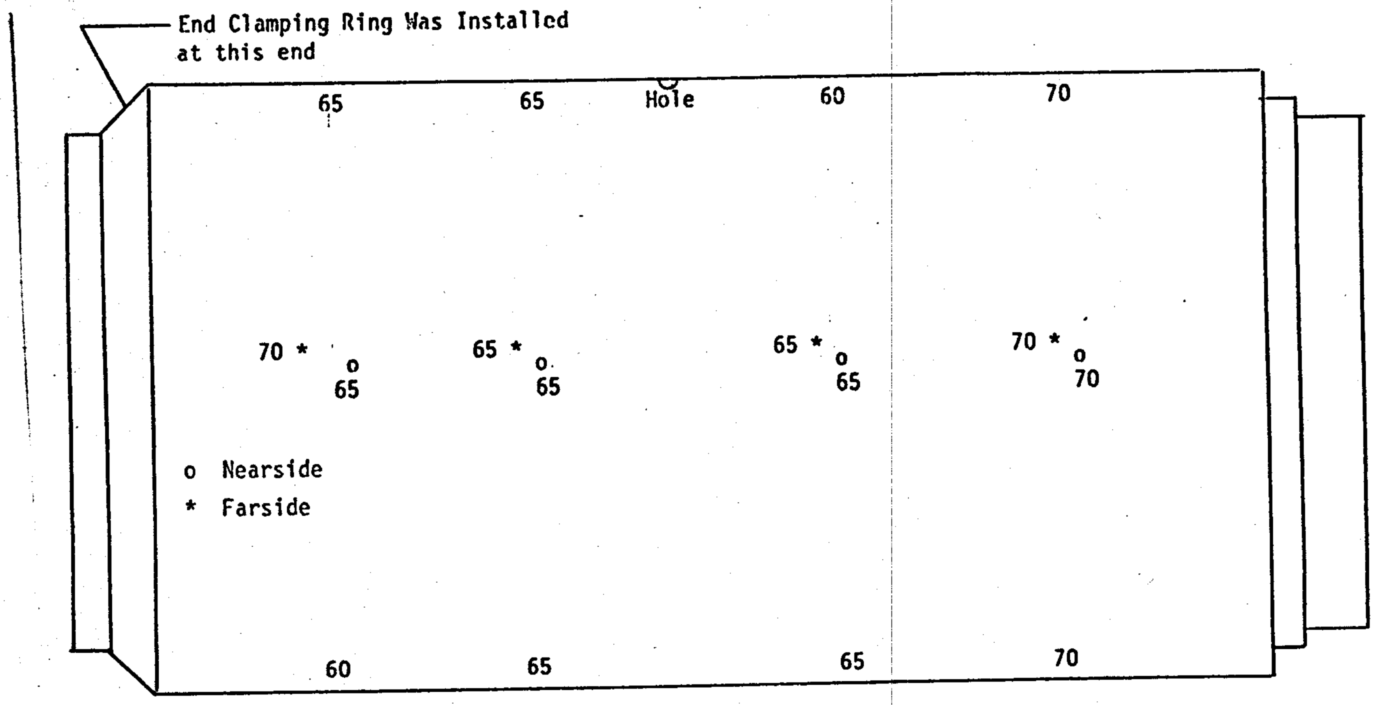
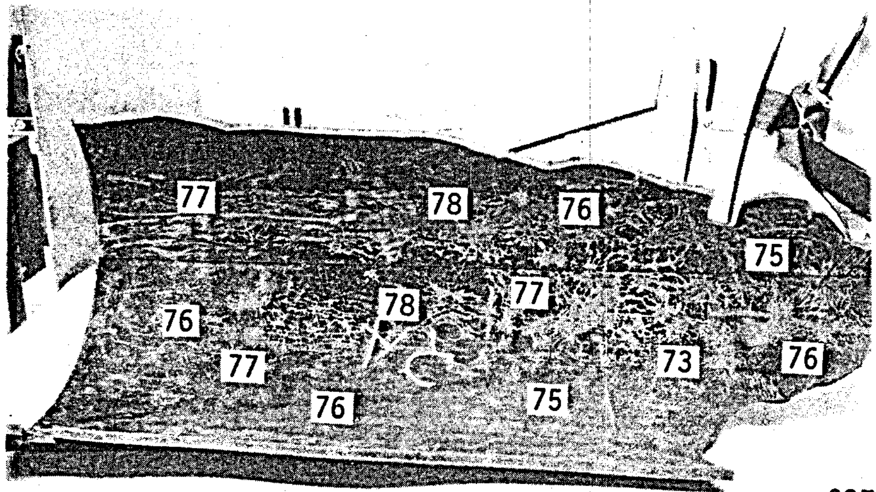


Figure 11. Pre Field Test Shore A Hardness Measurements

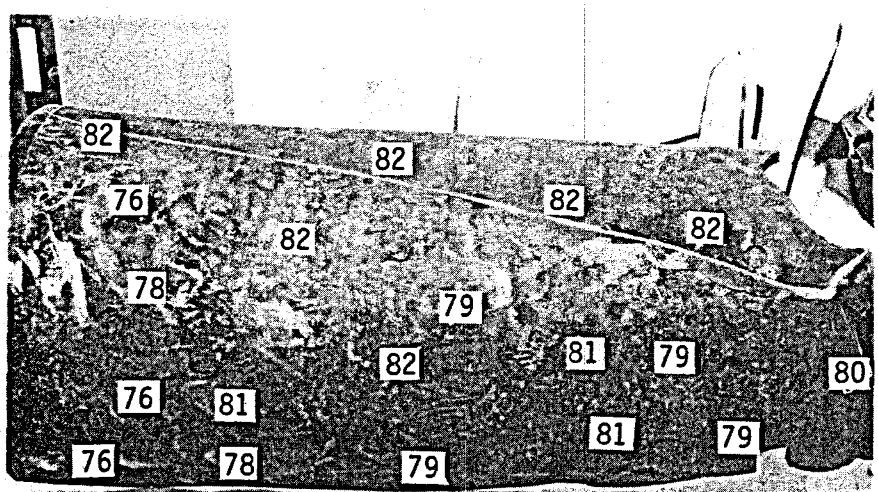
Once the post mortem parts were retrieved from the test site, Shore A hardness measurements were made on the two major pieces on both the OD and ID surfaces. Measurements were at points distributed over each surface as shown in Figures 12 and 13. A summary of those measurement follows in Table XII:

TABLE XII. SUMMARY OF POST FIELD TEST HARDNESS MEASUREMENTS

Part	Side	No. Data Pts.	Shore A Hardness Mean	Shore A Hardness Std. Deviation
2A	OD	19	79.7	± 2.18
2A	ID	12	76.2	± 1.40
3B	OD	9	71.9	± 5.25
3B	ID	9	73.4	± 1.42

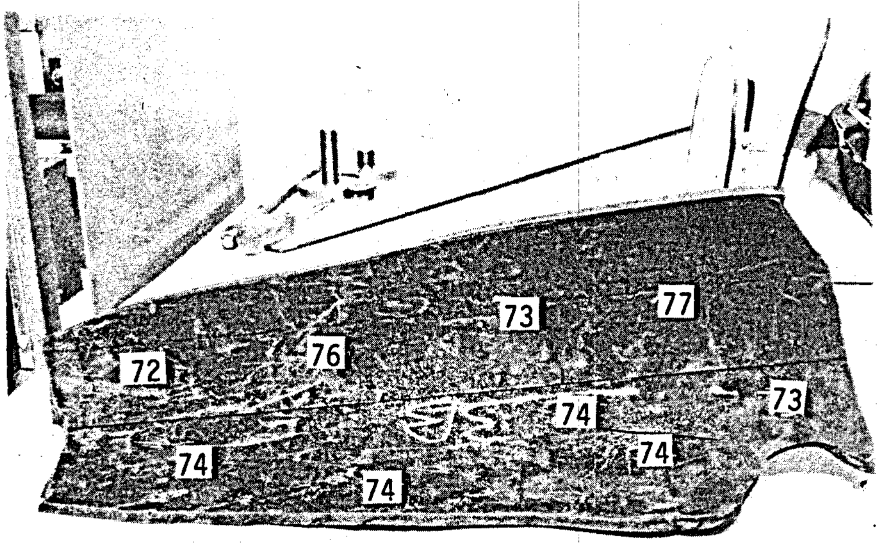


INSIDE DIAMETER



OUTSIDE DIAMETER

Figure 12. Postfieldtest Shore A Hardness Measurements, Part 2A



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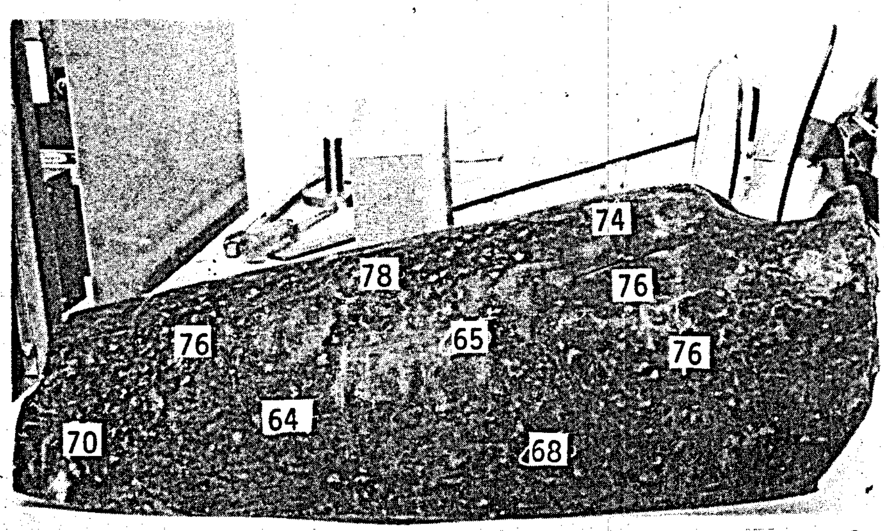

OUTSIDE DIAMETER

Figure 13. Postfieldtest Shore A Hardness Measurements, Part 3B.

It can be seen from these data in this subsection that there was a general hardening from about 66 Shore A to the range of 72 to 80 during the field exposure. This general hardening was anticipated and is due to in situ postcuring. The in situ postcuring differs from the standard Y267 EPDM postcuring in the following four significant ways: brine environment vs nitrogen, a minimum pressure of the saturation pressure of the brine vs atmospheric pressure, a lower temperature than the Y267 EPDM process level of 288C (550^{o}), and a different temperature-time history. Consequently, the degree of extraction of lower molecular weight substances from the Y267 EPDM and the extent of crosslinking would differ. The effect of the field exposure was somewhat different between Part 2A and 3B. Part 2A was somewhat harder with the OD side a little harder than the ID side with no apparent trend with 3B. It was not apparent that the local or piece to piece differences had any significant meaning.

4.6 FIELD EXPERIMENT CONCLUSIONS

Several anomalies existed in the field test, some which were known and accepted, and others that occurred and were beyond the control of the experiment. However, several important facts were learned from the experiment.

4.6.1 Experiment Anomalies

As discussed in paragraph 4.4.2B, it was decided to field the Y267 EPDM liner prototype knowing that imperfect adhesion of the liner to the metal tubing existed and the postcure was not completed. A pinching ring was devised and installed by Unocal to help compensate for the nonadhesion. The choice existed between fielding the imperfect prototype and not fielding anything. It was judged that any information gained would be better than none.

The second anomaly is associated with the fact that the liner was torn from the tubing. Consequently the liner underwent relatively violent mechanical stresses which a nominal liner would never see.

The third anomaly is associated with the test conditions. Before the system was up for any extended time, there were at least three up and down cycles which lasted from 1.5 to 2.5 hours. The longest sustained run was about 11 days. The duty cycle anomalies are judged not to be nearly so significant as the anomalies associated with the prototype.

4.6.2 Experiment Conclusions

Several things were learned about testing a prototype liner such as this, and some of the following conclusions were made to help the next experiment to be more fully successful.

A. Adhesion problems were encountered in scaling-up from laboratory coupons to tubular prototypes. A major difference which was probably significant was that the coupling system was applied as a liquid and the prototype had a full spectrum of surface orientations -- vertical to horizontal face-up and face-down versus the coupon which was horizontal face down.

B. The liner prototype and stinger configuration worked well and provided a practical way to test the liner, and insert and mount it into the flow stream.

C. Nonadhesion is reason for concern because gasses will tend to collect in the space behind the liner and when the line pressure falls off as the brine supply is shutdown the trapped gas will tend to balloon out the liner as the differential pressure reverses.

D. The pinch ring used to clamp the end of the elastomer in place worked well, however, with any nonadhesion a ring to clamp both ends is recommended.

E. The coupling system provided significant corrosion protection to the steel corrosion specimen for the time, probably several days, it was exposed directly to the brine.

F. No conclusions can be drawn from this experiment as to the ultimate feasibility Y267 EPDM liner concept because of the various anomalies.

G. Bright uncorroded steel existed behind the pinching ring where the Y267 EPDM liner was held into place throughout the field exposure.

5.0 Y267 LINER FULL-SCALE PRODUCTION COST

A most pertinent question regarding the Y267 EPDM Liner concept regards its ultimate cost in full-scale production. This question is extremely pertinent to assessing the ultimate pay-off that Y267 Liners can yield.

Unfortunately at this point substantial uncertainties remained because the processes to fabricate the lined tubing were still in question. The methods required to achieve reliable Y267/metal adhesion needed further definition and the subject of end interfaces had not been explicitly addressed. However, some costs were known and were available. Those known costs were documented herein and provide a partial list of cost components in 1987 dollars. As more of the technology questions are answered, the list of cost components can be further completed, and ultimately a comprehensive cost will be developed.

Table XIII shows a summary of costs that were available as well as those which were not. It shows that Y267 lined 8-5/8 inch steel tubing cost $27.17 per meter ($8.28/ft) plus tooling, capital costs, overhead, and profit. If high alloy termination rings were needed, an additional $21.33 per meter ($6.50/ft) would be added assuming 9.1 m lengths.

TABLE XIII. Y267 LINED TUBING SUMMARY BREAKDOWN OF COSTS

	$/m	($/ft)	$/m	($/ft)
Y267 EPDM Lining			27.17 +?	(8.28 +?)
Raw Materials	11.75	(3.58)		
Processing	11.12	(3.39)		
Scrap	2.53	(0.77)		
Labor	1.77	(0.54)		
Tooling	?	(?)		
Cap. Costs, OH, Profit	?	(?)		
Steel Tubing, 8-5/8, 36 ppf			35.43	(10.80)
Total Basic Costs			62.60 +?	(19.08 +?)
High Alloy Rings @ 195 $/set, 9.1 m (30 ft) lengths			21.33	(6.50)

All of the above costs were based on a 6.35 mm (0.25 in) thick Y267 liner in 8-5/8 in. 36 ppf tubing, the same as the prototype which was built. The cost of the raw Y267 materials was 11.75 $/m (3.58 $/ft), and the mixing, calendering, and scrap was estimated to cost 13.65 $/m (4.16 $/ft). If a high alloy interface ring is needed at either end of a 9.1 m (30 ft) length, the associated cost was estimated at about $195.00 or 21.33 $/m (6.50 $/ft). With the proper equipment it was estimated that two men could fabricate 8-9.1 m (30 ft) lengths of tubing per shift. This results in a direct labor cost of 1.77 $/m (.54 $/ft).

Optimization of various parameters remain for Y267 lined tubulars. The thickness of the Y267 EPDM liner and the steel tubing may be more cost effective at thinner dimensions. Less Y267 may provide adequate protection and thinner wall tubing would be possible since less sacrificial wall thickness for corrosion purposes would not be necessary. An important goal is to develop terminations that are integral with the Y267 lining and made from Y267 EPDM. This would preclude the need for expensive high alloy termination rings.

At this writing there were several significant cost factors which were uncertain and needed further definition to enable estimation of the overall costs for Y267 lining. More development to adapt the Y267/steel coupling system to tubular configurations and optimization of the design from the standpoint of life cycle cost effectiveness remained.

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