SERVICE LIFE PREDICTION OF RUBBER PARTS USED IN ENGINEERING APPLICATIONS - CASE STUDIES

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INTRODUCTION

The topic of Service Life Prediction is of both practical and scientific interest. The service life of a elastomer sets limit to engineering design. Therefore, Life Prediction of elastomers should be part of the engineering design process. Elastomer properties are sensitive to heat, moisture, light, fluids and mechanical stress (Figure 1). Elastomers can undergo changes in properties large enough to cause product failure. Most elastomer parts, in engineering applications, are intended to be in service for several years. Hence, the engineer or the developing scientist cannot wait that long to evaluate the aging process in actual service conditions. The three major engineering tasks in elastomer applications are to determine the Shelf Life, Service Life, and Remaining Useful Life (Part already in use).

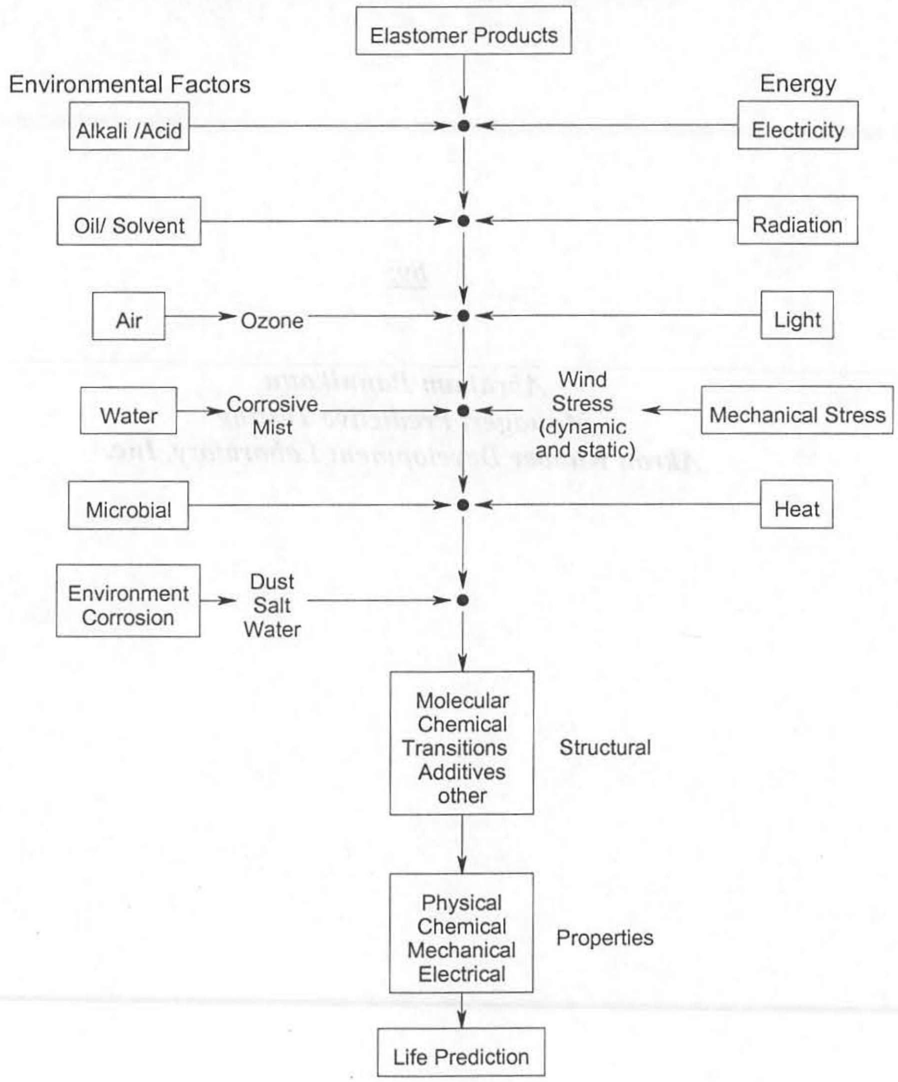

Figure 1

Elastomeric materials are frequently used under severe thermal, chemical and mechanical stress conditions (Table 1). A major drawback of elastomers is their tendency to oxidize. Most of them become unstable in contact with atmospheric oxygen. Elastomers are also affected by low and high temperature. The wide range of service conditions produces changes in physical, chemical, thermodynamic and other properties of elastomers(Table 2). These service conditions can change in an uncontrolled manner.

Comprehensive Service Life Prediction studies should involve fundamental changes in physical properties of the material due to degradation mechanisms, inter-atomic bonding, microstructure and crystal structures. Elastomer degradation should also include characteristics such as the loss of plasticizer, separation of polymer from fillers, and surface friction.

Service Life Prediction is clearly of great scientific interest and has attracted the attention of chemists, engineers, scientists and solid state physicists. Hence, it is not surprising that the topic has historically been discussed form a variety of different scientific approaches. There is a great need for a comprehensive Life Prediction model which incorporates various approaches. One of the main challenges in developing a service life is that several parameters to be considered both from the material and service environment. Many of these parameters are difficult to describe precisely in a mathematical model. The objective of this paper is to establish a practical usable method for quantitative life prediction of elastomers. This paper describes methods of Service Life Prediction for an O-ring, a Fiber-reinforced Rubber Pipe Joint and a Conveyer Belt Cover.

Table 1: Main Factors Responsible for Degradation.

Thermal	Mechanical
Thermo-Oxidative	Hydrolytic
Photo	Chemical
Photooxidative	High Energy Radiation
Ozone	

Table 2: Dominating Events during Degradation.

Random Chain Scission	Substitution
Depolymerization	Plasticizer Loss
Crosslinking	Filler Bonding Change
Side Group Elimination	

Akron Rubber Development Laboratory, Inc. (ARDL) Methodology for Quantitative Life Prediction of Elastomers

Quantitative Service Life Prediction of the elastomeric component is becomes an increasingly important requirement as elastomers are used for more critical engineering applications. Service life prediction methodology should include all processes that may affect the function of the elastomeric component. ARDL's approach is to: 1., select the predominant degradation processes and establish an appropriate accelerated aging test; 2., compare the failure mode/degradation process of lab samples with field sampling using chemical, physical and optical techniques (Table 3); 3., establish the failure rates using accelerated lab tests; and 4., extrapolate rates to the service condition to determine service life.

Table 3: Techniques

Optical Microscope
Video Microscope
Scanning Electron Microscopy
FT-IR
GC/MS
DSC
DMA
Pulse NMR
TGA
Crosslink Density (Wet Chemistry)
Specific Gravity
Micro Hardness
Micro Hardness Decay
Dielectric Constant
Electron Spin Resonance

ARDL's methodology flowcharts are shown in Figures 2a and 2b. The first step is to define the function(s) of the elastomer component. Based on the function, establish a failure criterion. The failure criterion may be an unacceptable change in function and the change may cause a particular failure. Changes may be stress relaxation, creep, tear resistance, stiffness/modulus change, swelling, dielectric properties, dynamic proprieties, etc. Then, characterize and identify the underlining mechanism involved in this change. Establish the rate of change by accelerated laboratory test at different levels of severity and at different time intervals. It is important to keep the accelerated test condition similar to the service condition and perform the test at four temperatures higher than average service temperature.

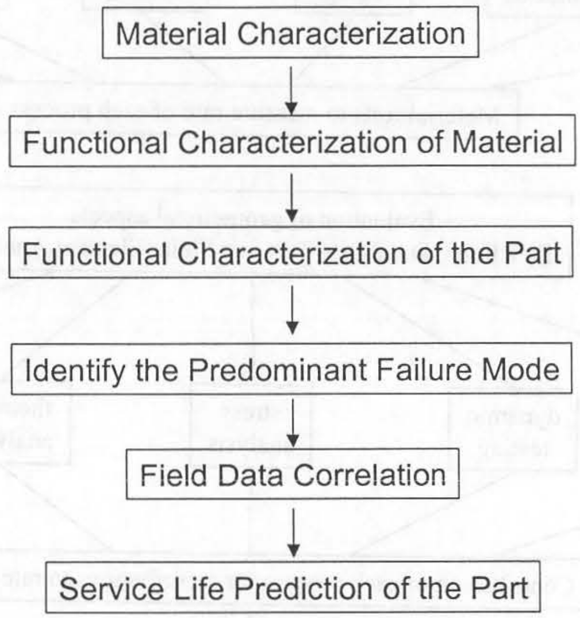

Figure 2a

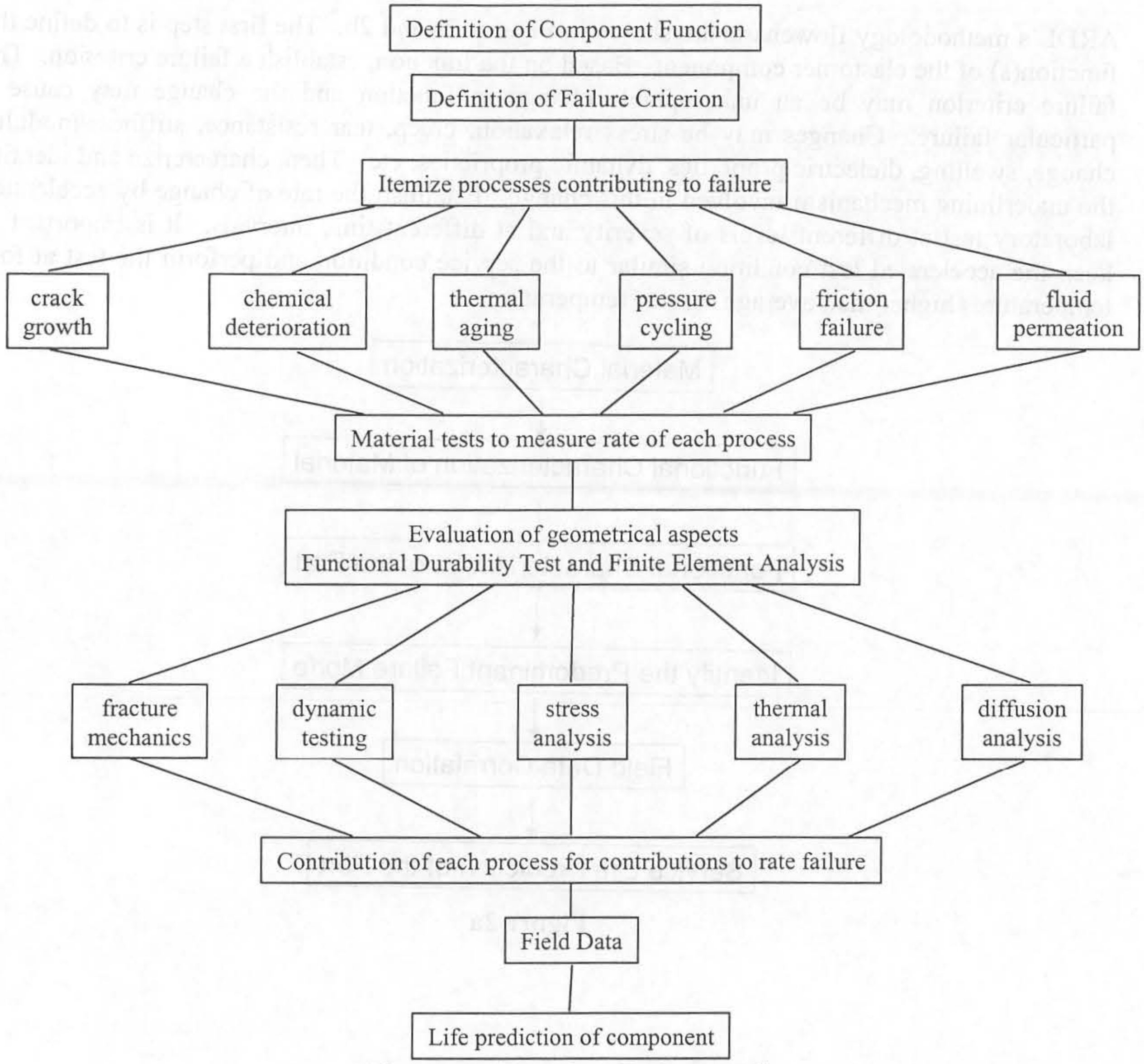

Figure 2b (Based on Reference 6)

Aging process in the accelerated test should be verified with "service aging" process. The verification can be done by chemical or physical evaluation of both field samples and samples from different levels of accelerated test conditions and time intervals.

After the rate of degradation process has been determined using accelerated laboratory testing, then the rate at the service temperature can be determined by Arrhenius extrapolation or by time-temperature superposition extrapolation. It is important to study the mechanism(s) of failure in service and correlate with each accelerated aging test. This will help to improve reliability of the Service Life Prediction Model.

Arrhenius Approach

This approach of life prediction involves determining the rate of failure for a range of temperatures of interest and then plotting these rates on an Arrhenius type plot against reciprocal absolute temperature. This approach considers the failure process as a chemical reaction where the rate of reaction will increase as temperature increases.

$$\text{Service Life} = Ae^{\frac{E_a}{RT}}$$

Where:

E_a is the activation energy for the failure process

A is a constant depending on type of failure

To be able to use Arrhenius equation, accelerated testing should be used to identify the "time-to-failure" for a minimum of four temperatures above the average service temperature. Service life prediction can be obtained by plotting the logarithm of the "time-to-failure" versus the inverse of absolute temperature ($^1/_T$) and extrapolating the curve such that it intersects the straight line $y = {}^1\!/_{T_{sevice}}$ ($T_{service}$ is the average service temperature). To estimate the expected time-to-failure, it is important to use a material property which features sufficient range to assure a reliable determination at any particular stage of the property during both accelerated aging and field aging.

Time - Temperature Superposition Approach

This approach considers the rate of deterioration as a predominantly viscoelastic behavior. This approach will allow the investigator to develop a master curve for service life prediction using the WLF-equation (Williams, Landel and Ferry) the general form of which is:

$$\log a_T = \frac{C_1(T - T_s)}{C_2 + (T - T_s)}$$

Where:

T_s is reference temperatures (usually T_s should be 50K above T_g)

a_T is the shift factor

C_1 and C_2 are material constants (often $C_1 = 17.44$ and $C_2 = 51.6$)

T is the test temperature

From the present point of view, it must be noted that by rule of thumb one decade of test time increase is equivalent to an increase in temperature of approximately 6-7°C. (The main problem with this approach is that polymer degradation may not follow strict viscoelastic behavior.) The equation permits the shifting of data taken over a time range and at a variety of temperatures along the time axis, to create a master curve which extends the time axis by several order of magnitude.

CASE STUDIES

Case 1: Life Prediction of O-Rings by Compression Stress Relaxation Testing

The two important properties of elastomeric seals are Compression Stress Relaxation and Dynamic Response. Dynamic Response is a measure of the ability of the seal to retract to its original shape after a constraint has been removed. Rapid recovery of an elastomeric seal is important in which the contact between the seal and mating surface is momentarily broken due to vibration or dynamic motion. Dynamic properties of elastomers can be measured using force dynamics. The force decay of elastomer components under constant compressive strain is known as compression stress relaxation. The test measures the sealing force exerted by a seal or O-ring under compression between two plates (Figure 3). This property is very important where seals are relatively inaccessible, and the cost of replacement is too high. In such instances, life prediction is highly desirable. The automotive industry has focused on the percent of retained sealing force as a function time in a compression stress relaxation test.

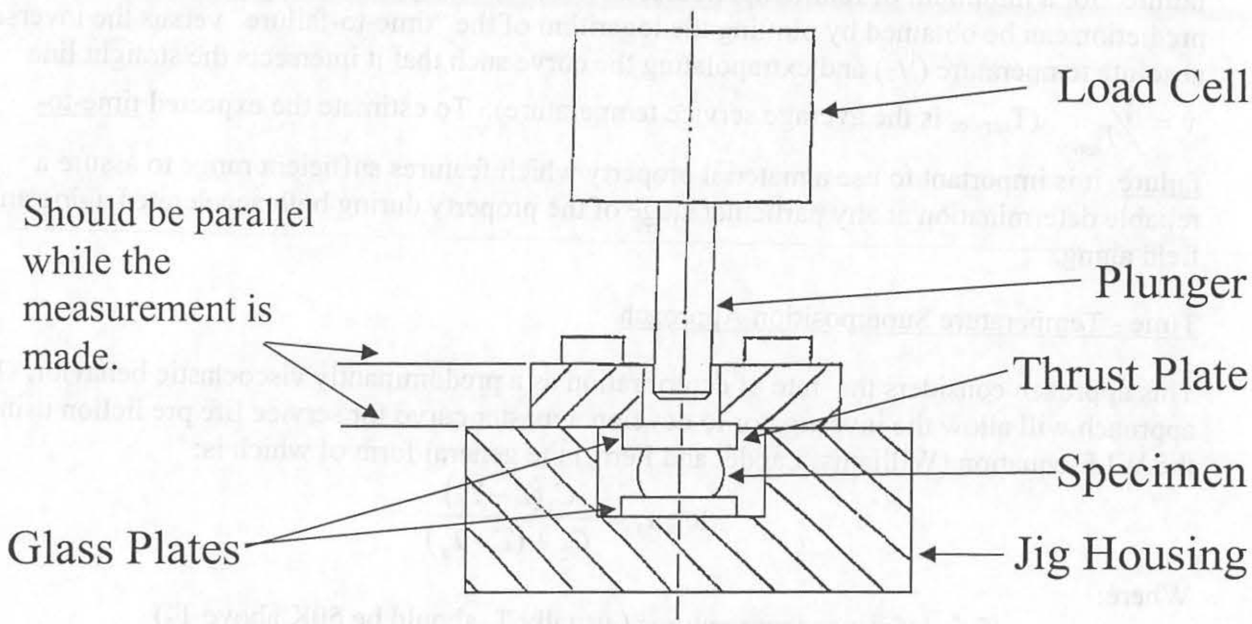

Figure 3

The apparatus used at ARDL to study compression stress relaxation is the Wykeham-Farrance compression stress relaxation equipment. Cylindrical samples are used in accordance with ISO 3384. Compression stress relaxation is used for the service life prediction by measuring the sealing force decay as a function of time and temperature. IRM 903 is used as the aging fluid. A cross-sectional view of the test jig is shown in Figure 4. These curves are obtained at 25 percent constant compressive strain at three accelerated aging temperatures (100ºC, 125ºC and 150ºC). Sixty percent sealing force decay is selected as the failure point. This failure point is developed based on an earlier study by ARDL using Leak test, Compression Stress Relaxation testing, Dynamic testing and Automotive OEM Engineering data.

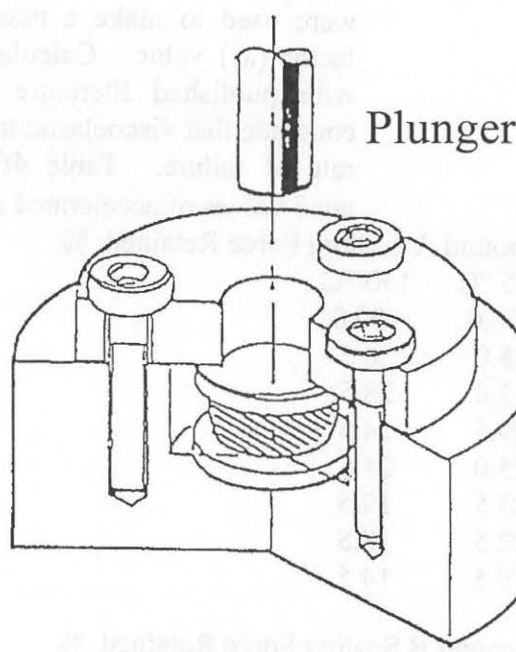

Figure 4

Field data was collected for silicone compound A, B and C. Chemical and physical characterization of field sample and lab samples was used to verify the mode of failure and degradation process.

<u>Test Parameter</u>

Test:	Compression Stress Relaxation	
Accelerated Aging:	Accelerated oven aging at 100ºC, 150ºC and 150ºC	
Aging Fluid:	IRM 903	
Test Condition:	Sample were aged inside jigs with 25% compression	
Failure Mode:	Stress Relaxation	
Failure Criteria:	Compress Stress Relaxation (Force Retained, %)	40%
	Dynamic Properties	
	tan δ	20% increase max
	dynamic modulus	80% increase max
Extrapolation Techniques:	Arrhenius and WLF	
Results:	Shown in Tables 4d and 4e based on Arrhenius extrapolation technique. The data in Tables 4a, 4b and 4c	

were used to make a master curve to determine a shift factor (a_T) value. Calculated values were not consistent with published literature values. From this one can conclude that viscoelastic mechanisms do not determine the rate of failure. Table 4f shows dynamic modulus and tan δ values of accelerated aged samples.

Table 4a: Silicone Compound A Sealing Force Retained, %

hours	100 °C	125 °C	150 °C
0.5	100.0	100.0	100.0
48	40.5	38.0	36.5
168	34.5	33.0	28.5
336	31.0	29.5	24.5
840	29.5	25.0	21.5
1008	28.5	23.5	19.5
2000	26.5	22.5	18.5
5000	25.0	19.5	14.5

Table 4b: Silicone Compound B Sealing Force Retained, %

hours	100 °C	125 °C	150 °C
0	100.0	100.0	100.0
48	81.0	75.5	71.5
168	69.5	62.5	56.5
336	62.5	56.0	48.0
840	58.0	51.0	44.0
1008	56.0	48.0	40.0
2000	54.0	45.5	36.5
5000	48.0	40.0	26.5

Figure 4c: Silicone Compound C Sealing force retained, %

hours	100 °C	125 °C	150 °C
0	100.0	100.0	100.0
48	61.0	56.0	53.5
168	52.5	47.5	42.5
336	48.0	41.5	36.5
840	42.0	38.0	32.5
1008	41.5	35.5	29.5
2000	40.0	34.0	27.0
5000	37.5	28.5	20.5

Table 4d:

Property	A	B	C
Hardness, Shore A	59	55	64
Ultimate tensile, MPa	5.8	4.7	6.9
Compression Set, %			
22 hrs/70°C	33	16	14
22 hrs/100°C	73	18	17
22 hrs/125°C	87	26	22
22 hrs/150°C	89	34	35
Sealing Force Retained, %			
168 hrs/150°C	28.5	56.5	42.5
1008 hrs/150°C	F	48.0	29.5
Field Data	F*	G	F**

*immediately **after 1 year service

Table 4e:

Compound	A	B	C
Field Data	Early	5 years +	1 year
Extrapolated Arrhenius	61 hours	6 years	7 months
WLF	Not Successful		

Table 4f: Dynamic Modulus by DMA (tan □)
150°C

Compound	A	B	C
0	10.5 (0.22)	9.5 (0.20)	12.5 (0.24)
48	18.9 (0.25)	10.8 (0.21)	13.8 (0.25)
168	19.2 (0.26)	11.2 (0.21)	16.8 (0.27)
336	19.8 (0.27)	12.8 (0.22)	22.8 (0.28)
840	20.4 (0.28)	14.8 (0.23)	24.8 (0.29)
1008	20.4 (0.28)	16.9 (0.24)	25.8 (0.30)

Case 2: <u>Remaining Useful Life Determination of Elastomeric Pipe Joints</u>

In this study, Arrhenius extrapolation technique with Laboratory accelerated oven aging testing was used to establish the Remain Useful Life of Elastomeric Pipe Joints. Failure mode of this part was identified as oxidative degradation.

Test Parameter

Accelerated Aging Test:	Accelerated oven aging at 60°C, 80°C and 100°C
Field Samples:	a) Inside section b) Middle section c) Outside section
Service Duration:	Five years
Average Service Temperature:	45°C
Measurement Temperature:	DSC induction time measurement
Failure Mode Evaluation:	Dynamic testing - Damping coefficient (N-sec/mm)

Differential Scanning Calorimetry (DSC) ASTM D-3418-88

The sample was heated from 30°C to 300°C at 25°C per minute in helium for approximately 10 minutes. The sample was allowed to equilibrate for 6 minutes. After equilibrating, the gas purge was switched from helium to oxygen. DSC induction time was calculated based on time from introduction of the oxygen to completion of decomposition.

Induction Time Of Field Samples:
1) Outside layer = 28 minutes
2) Middle section = 44 minutes
3) Inside layer = 38 minutes

Damping Coefficient (N-sec/mm) of field sample:
1) Outside layer = 38.88
2) Middle section = 2.57
3) Inside layer = 16.43

Failure Point: Failure point is calculated based on failed field sample. (Induction time = 13 minutes and Damping coefficient = 51.5 N-sec/mm)

Service Life Prediction Extrapolation Technique: Arrhenius Approach

Table 5a: Induction Time by DSC

hours	100 °C	80 °C	60 °C
0	45	45	45
4	35	41	42.5
16	18	32	36
48	6	25	34
168	0	17	30
1000	0	4	24
2000	0	2	19

*Extrapolated service life – 8 years. Therefor the remaining useful life of this part will be 3 years.

Table 5b

		Induction Time minutes	Damping Coefficient N-sec/mm
Part in Use	Inside	38	16.43
	Inner Case	44	2.57
	Outside	28	38.88
Failed Part	Outside	13	51.5

Table 5c: Damping Coefficient by DMA (N-sec/mm)

Teperature	100°C	80°C	60°C
0	1.58	1.58	1.58
48	68.1	28.4	16.5
168	98.1	52.8	24.5
2000	failed	108.5	48.7

Case 3: Service Life Prediction of Industrial Conveyer Belt Covers:

Heavy, steel cable-reinforced conveyer belts are commonly used in the coal industry. The failure mode of the belt cover in the particular application was identified as hydrolysis with subsequent ultimate tear failure.

Test Parameter

Accelerated Test:	Fatigue test at 10 Hz with 10% dynamic tension test
Aging Fluid:	Water
Test Temperature:	60ºC, 80ºC and 100ºC
Test:	Five million cycles
Measurement Temperature:	MTS Dynamic characters on dumbbell sample at 5 and 2
Properties:	Damping coefficient
Average Service Temperature:	40ºC

Table 6a: Cycles to Failure

Temp	Comp A	Comp B
100	120000	180000
80	700000	1000000
60	2000000	4000000
Extrapolated	4,600,000	9,200,000

Table 6b: Damping Coefficient at 60 by DMA (N-sec/mm)

	Compound A	Compound B
0	0.852	0.684
1 million	1.048	0.845
2 million	1.284	1.048
3 million	0.748	1.128
4 million	failed	1.232
5 million		0.848
6 million		failed

CONCLUSION

The above approaches can be applied to determine life of elastomers components used in engineering applications. However, it is important to define failure mode and failure mechanism. It is also important to establish verification and correlation between field and lab samples using physical and chemical techniques. The primary rate determining mechanism of component failure can be predicted using the Arrhenius methodology. The Arrhenius method provides a quantitative determination of the service life of elastomer components in a particular application. Further research studies are required for each new application. The service life prediction effort conducted on elastomeric materials provides a good materials database for computer-aided design engineers who in turn can use the information to effectively model part durability, thus reducing the need for complex and costly prototype testing.

Service life prediction as an engineering tool is still in the infancy stage of development. Although some successes have been reported, more application research is needed. Recent and continuing interest showed by the automotive OEM's is likely to lead to further advances in elastomer service life prediction.

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APPENDIX

Final Draft of ISO/FDIS 11346: Rubber, vulcanized or thermoplastic – Estimation of life-time and maximum temperature of use form and Arrhenius plot. This draft is attached for review and comment related to the preparation of this document. Forward your comments to Abraham Pannikottu.