DE-Vol. 100, Reliability, Stress Analysis, and Failure Prevention Aspects
of Adhesive and Bolted Joints, Rubber Components,
and Composite Springs
ASME 1998

Performance Prediction of Elastomeric Gasket Materials by Compression Stress Relaxation

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ABSTRACT

In the automotive industry, the most widely used technique to predict the sealing capacity of an elastomeric gasket material is based on compression set data which is obtained after short term aging in air or reference fluids. Compression set measures the percentage of permanent deformation after the removal of the applied strain. This is an indirect measure of the sealing capacity and it can easily be misleading as a predictor of the sealing performance of gasket materials. Recent increases in service temperature requirements along with use of "SG" grade motor oils have created the need for a better means of assessing the high temperature, long term performance of elastomeric materials used in automobile engine gaskets. This paper reports Compression Stress Relaxation measurements on a variety of gasket materials using the Wykeham-Farrance Compression Stress Relaxation tester. This study also examines the effect of cycling heating and cooling on the retention of sealing force of gasket materials and includes the cumulative effect of exposure of the gasket material to motor oils and ASTM #2 oil at 125°C up to 1008 hours.

INTRODUCTION

The stress decay of rubber components under constant deformation is known as stress relaxation. When an elastomeric part is subjected to a specific strain and maintained over time, the internal stress developed in the material decays exponentially with time from the initial maximum to an eventual equilibrium state. This relaxation phenomenon is due to physical macromolecular rearrangement and chemical chain scissions.1 The extent and the rate at which such relaxation occurs is critical to performance in certain important rubber and plastic applications. For example, in gasket sealing applications, the extent of compression stress relaxation determines the effectiveness of the gasket in maintaining a good seal.

In the rubber industry, the most widely used technique to predict the sealing capacity of an elastomer is compression set (ASTM D 395, Method B). Compression set testing measures the percentage of permanent deformation after the removal of a constant static strain. This is an indirect measure of sealing capability. Furthermore, the recent increases in underhood temperatures along with use of "SG" grade motor oils have created a need for better methodology for estimating the high temperature performance of elastomeric gasket and sealing materials. This paper reports long term compression stress relaxation measurements on a variety of oil-resistant rubber materials and also examines the effect of exposure on the materials to aerated motor oils at 125°C.

EXPERIMENTAL

The rubber materials evaluated were Silicone Rubber (PVMQ), Fluoroelastomer (FKM), Polyacrylate (ACM) and Hydrogenated Nitrile Rubber (HNBR). The rubber formulations were specifically compounded for gasket applications. The formulations are specified in Tables 1-4.

Table 1

SILICONE RUBBER

Ingredients:	
Silastic® TR-70	50.0
Silastic® NPC-40	50.0
Silastic® HT-Modifier	1.0
AN-3	3.0
STI®-V	1.0
Press cured 10 min. @ 177°C	
No Post Cure	

Table 2

FLUOROELASTOMER

Ingredients:	
Viton® E-60	100.0
Maglite® D	3.0
N990 Black	30.0
Calcium Hydroxide	6.0
Curative #20	1.8
Curative #40	4.0
Press cured 10 min. @ 177°C	
Post Cured 24 hrs. @ 232°C	

Table 3

POLYACRYLATE

Ingredients:	
Hytemp® 4051	100.0
N550 Black	65.0
Stearic Acid	1.0
Struktol® WB212	2.0
Agerite Stalite S	2.0
Sodium Stearate	4.0
Hytemp® NPC 50	2.0
Press cured 10 min. @ 190°C	
Post Cured 4 hrs. @ 177°C	

Table 4

HNBR

Ingredients:	
Zetpol® 2010	100.0
N990 Black	30.0
TOTM*	5.0
Maglite® D	10.0
Naugard 445	2.0
Vanox ZMTI	1.0
HVA #2	7.5
Press cured 30 min. @ 170°C	
Post Cured 4 hrs. @ 150°C	

* Trioctyl Trimellitate

Material properties on each compound were characterized by hardness, crosslink density, volume swell, and compression set after proper curing and post vulcanization (Table 5). Stress Relaxation measurements were made on cylindrical specimens, 13.0 ± 0.8 mm in diameter and 6.3 ± 0.4 mm in thickness.

Table 5

ORIGINAL PROPERTIES OF TEST ELASTOMERS

Properties	PVMQ	FKM	ACM	HNBR
Hardness, IRHD[a]	55	76	72	68
Crosslink Density, $x10^{-4}$ moles/cm^3	1.51	7.68	1.51	2.28
Volume Swell[b]:				
ASTM #2 Oil	6.8	1.5	-0.7	3.3
5W30	29.6	1.1	0.1	2.9
15W40	19.3	1.1	0.2	2.4
Compression Set, %[c]	10.8	5.9	10.8	19.1

[a] ASTM D 1415-88
[b] ASTM D 471-91
[c] ASTM D 395-89

Compression stress relaxation measurements were performed using the Wykeham-Farrance compression stress relaxation equipment (Figure 1). The equipment consists of force measuring device and jigs to maintain the specimen at constant strain. The force measuring device is used to periodically monitor the counterforce exerted by the specimen. The jigs, which hold the cylindrical specimen can be maintained under various environmental conditions. A more detailed discussion of test equipment and principles is given in references 2 and 3.

This testing program was designed to be consistent with Method A of ISO 6056-1987 and BS 903 Part A42-19833. A compressive strain of 25% was applied and counterforce measurements were made both at room temperature and 125°C. At room temperature, the specimen was compressed to 25% strain within a 30 second period. The jib was then half filled with fluid, partially immersing the sample, an placed in the oven for one hour at 125°C. After this initial aging, at 125°C, the sealing force measurement was made. The jig was then cooled to room temperature and the initial room temperature sealing force measurement was made. In the same manner, subsequent sealing force measurements were made at 125°C and room temperature after completion of 24, 72, 168, 336, 504, 840, and 1008 hour time intervals in the aging oven at 125°C. The jig was refilled to half level with fresh oil after each measurement.

All four compounds were tested in three different oils and in air at 125°C. The three oils were ASTM #2, 5W30 SG/CD Pennzoil Q, and 15W40 SG/CF-X Shell Super 2000 Rotella T.

RESULTS AND DISCUSSION

Figures 2-5 summarize the stress relaxation test results of four compounds run in triplicate in four environments at 125°C. All the percentage retained force values are calculated based on one hour aged initial reading.

Table 6

Absolute Sealing Force Retained After Exposure To Various Environments @ 125°C

		HOT AIR		ASTM #2 OIL		5W30		15W40	
	RT	125°C	RT	125°C	RT	125°C	RT	125°C	RT
PVMQ	I[a]	99.2	170.4	90.3	176.6	83.2	149.0	88.1	137.9
	F[b]	47.6	71.6	29.8	59.6	24.5	38.7	17.8	31.6
FKM	I[a]	172.1	270.0	173.5	300.2	150.3	262.4	162.4	287.0
	F[b]	96.5	134.8	110.8	186.4	94.3	175.7	110.5	183.7
ACM	I[a]	128.1	204.6	139.2	241.1	132.6	227.7	123.7	195.7
	F[b]	66.7	84.5	93.4	153.5	83.8	129.0	77.8	123.2
HNBR	I[a]	149.9	241.1	154.3	291.3	159.2	275.8	145.9	248.2
	F[b]	55.6	83.2	70.3	155.2	52.9	120.1	52.5	110.3

[a] Initial Sealing Force, Newtons (N), after 1 hour aging

[b] Final Sealing Force, Newtons (N), after 1008 hours aging

The results in Table 6 indicate that the absolute sealing force values measured at 125°C were approximately 1.7 times those of room temperature under all conditions. The polyacrylate and HNBR compounds performed better in oil than in hot air. The Silicone compound performed better in hot air than in oil. As expected, the ASTM #2 oil appeared to be least aggressive of the three oils. The 5W30 oil was the most aggressive. Statistical analysis of the data indicates that the coefficient of variation for a single measurement was found to be ±3% in hot air, while that of the measurement taken in oil was found to be ±6%.

CONCLUSION

Compression stress relaxation testing is an important method for assessing the sealing performance of gasket materials in oil at elevated temperatures. With appropriate conditions, the test is sufficiently reproducible. For comparative performance ratings among materials, both a minimum retained force as well as a minimum percentage retained force should be required. We would recommend utilization of compression stress relaxation as a tool for compound development for seal/gasket/o-ring materials.