

New Fluoroelastomer Developments For Aerospace Sealing Applications

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ABSTRACT

For almost half a century Viton[®] fluoroelastomers have performed successfully under the harsh and demanding operating conditions imposed by aerospace service. Extreme conditions of heat and cold are inherent to flight. Today, the vast majority of the world's military, commercial and business aircraft depend upon fluorohydrocarbon elastomers to maintain reliable, long life, leak-free performance.

This paper will review several new peroxide-curable fluorohydrocarbon elastomers capable of sealing from -45°C to +275°C in air, lubricating oils, and standard reference fluids. Evaluations will highlight fluid compatibility and seal force retention in gas turbine engine oils.

The new fluoroelastomers utilize Advanced Polymer Architecture designed to provide enhanced end-use properties, as well as significant improvements in processing behavior, which is essential in high quality, high yield fabrication of aerospace sealing devices.

Viton[®] fluoroelastomer is a registered trademark of DuPont Dow Elastomers L.L.C.

INTRODUCTION

The first commercial use for Viton[®] fluoroelastomer occurred in 1957 when the U. S. Air Force at Wright Field, Dayton, Ohio (now Wright-Patterson Air Force Base) was seeking more durable O-rings that had the ability to seal hot engine lubricants and hydraulic fluids. MIL-P-25897 was subsequently written around the capabilities of DuPont's Viton[®] A fluoroelastomer. This material displayed an unprecedented balance of heat and chemical resistance under the extreme exposure conditions imposed by aerospace. In the air, absolute sealing integrity is essential. Today, the vast majority of the world's military, commercial and business aircraft depend on the reliability and performance of Viton[®] fluoroelastomer.

Fluorohydrocarbon elastomers (FKM) are routinely used as sealing devices for gas turbine engines, auxiliary power units, air cycle machines, filters, integrated drive generators, hydraulic servos and actuators. Their performance properties have been well documented in many aircraft and missile components over the past 45 years. They include:

- O-rings used in fittings, connectors, valves, pumps, filters and oil reservoirs
- Radial lip seals used in pumps
- T-seals used in high pressure fittings and connectors
- Cap-seals
- Manifold gaskets
- Firewall seals
- Clips for jet engine wiring harnesses tire valve stem seals
- Siphon hose for hot engine lubricants

The evolution of gas turbine engine design is pushing fluoroelastomers to their thermal limits. This design progression encompasses a relentless drive towards more powerful, higher thrust, lighter weight, fuel efficient engines, with accompanying reductions in noise and emissions, as well as better reliability, operating safety and longer time on wing. These trends converge to push engine thermodynamics to their limits, invariably culminating in higher operating temperatures.

As a result, engine builders have adopted advanced lubricants with higher thermo-oxidative stability (HTS), in order to achieve long life engine performance. These HTS oils are significantly more aggressive towards standard fluoroelastomers based on vinylidene fluoride (VF₂) and hexafluoropropylene (HFP) than previous lubricants. As a result, there is a gradual shift from standard to specialty grades of fluoroelastomer that offer even higher performance. As the usage of fluoroelastomers has increased, aerospace seal fabricators have been driven to optimise part quality and manufacturing yields, in order to maintain competitive position, without sacrificing end-use properties.

Recently, DuPont Dow Elastomers has developed new polymerization and curing technology to manufacture specialty fluoroelastomers with end use properties that meet the evolving needs described. The new polymers are called Viton® made with Advanced Polymer Architecture, or simply Viton® APA.

The processing and end use properties of the new APA polymers are discussed more fully in the following text. Previous papers on the subject of APA technology have concentrated on experimental polymer data, however the data presented here is for the most part measured on polymer made in the new commercial manufacturing facility.

The fundamentals of standard and specialty types of fluoroelastomer and their compositions have been explained in detail in several previous publications [1,2,3,4,5,6,7,8] and just a brief explanation is given here to illustrate the positioning of APA.

Fluoroelastomers made with Advanced Polymer Architecture (APA)

Typically, fluoroelastomers are differentiated by their monomer composition and the cure system employed. Traditional dipolymer and terpolymer compositions, based on vinylidene fluoride (VF₂), hexafluoropropylene (HFP) and tetrafluoroethylene (TFE), are considered “standard” products within the industry. Other products offering some special aspect of functionality not provided for by the standard products are considered “specialty” types. This concept is illustrated by the examples shown in Figure 1. Here specialty products FKM-GLT and FKM-GFLT contain the monomer perfluoromethylvinylether (PMVE) to provide enhanced low temperature flexibility. While the basic building blocks (monomers) are the same, polymer architecture has advanced considerably. Today’s polymers tend to have superior properties and processing characteristics when compared to their predecessors. These advances in polymer design have been necessary to keep pace with the converters who are processing higher volumes of FKM articles with world-class efficiency and material utilization.

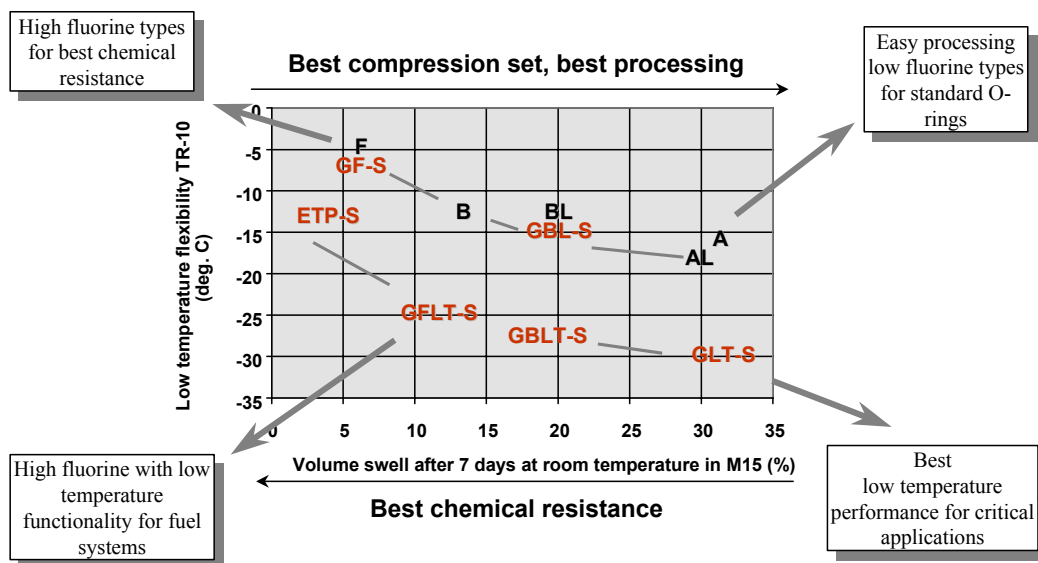


Figure 1. Standard and Specialty types of fluoroelastomer

Specialty types like FKM-GF-S and FKM-GBL-S contain in addition a cure site monomer to allow peroxide curing. The other group of polymers in Figure 1 all contain the monomer perfluoromethylvinylether (PMVE), which provides a significant improvement in low temperature flexibility and sealing capability. This paper concentrates on the specialty polymers FKM-GBL-S, GF-S, GLT-S, GBLT-S and GFLT-S. Details of the APA technology were first presented at the Fall ACS Rubber Division conference in 2001 [9,10]. Further discussion about the enhanced properties and processing benefits of these new specialty polymers were reviewed at the Spring International Conference on Fluid Sealing in 2003 [11].

The purpose of this paper is to review the new polymers made with APA technology and frame them within the context and needs of the aerospace market. The polymers are shown in Table 1:

Product Designation	Fluorine Content	Gum Mooney Viscosity (ML1+10/121°C)	Comparable Commercial Product
Viton [®] GLT-S (VTR-8500)	~65%	65	Viton [®] GLT, GLT-305
Viton [®] GBLT-S (VTR-8525)	~66%	65	Viton [®] GBLT-601
Viton [®] GFLT-S (VTR-8550)	~67%	65	Viton [®] GFLT, GFLT-301
Viton [®] GBL-S (VTR-8650)	~68%	65	Viton [®] GBL-900, GBL-200
Viton [®] GF-S (VTR-8600)	~70%	65	Viton [®] GF, GF-300

Table 1. Fluoroelastomers evaluated

These new polymers have a nominal 60-65 Mooney (ML 1 + 10 @ 121°C) and were originally developed for compression molding. Data on processing and end use properties will be presented to illustrate their performance.

The new fluoroelastomers made with APA technology have been designed to give an excellent balance of processing and physical properties. Compared with earlier peroxide cured types, the APA polymers show faster and more efficient curing with good mold flow, outstanding mold release and low mold fouling. These improvements are due to the modified molecular weight distributions, improved cure site monomers and unique end group chemistry of the new polymers.

Supply: At this time all the polymers discussed herein are available for sale in commercial quantities. The new polymers are being produced at DuPont Dow's new facility in Dordrecht, Netherlands. This facility commenced operations in the second quarter of 2002. These new products, with their respective VTR-designations, will be assigned permanent commercial names later this year.

EXPERIMENTAL

Sample Preparation:

Compounding of the fluoroelastomers was done using standard rubber lab equipment (internal mixer and two-roll mill.) Standard processing techniques commonly practiced in the industry were employed in preparation of test specimens. Compound formulations were typical of those recommended for O-ring sealing service. All contained 30 pph MT Carbon Black to produce a nominal 70-80 durometer hardness. The compound recipes are provided in Table 2 and the specific supplier of the compound ingredients is provided in Appendix I. Test specimens for all physical property and aging tests were die-cut from the molded slabs. Standard ASTM slabs and AS 568-214 O-rings were prepared by compression molding per the following conditions:

Curing Conditions:

- FKM-A-601C
 - Press Cure: 10 min/177°C Post Cure: 16 h/232°C in air
- FKM-GBL-200, GF, GFLT, GLT
 - Press Cure: 10 min/177°C Post Cure: 16 h/232°C in air
- FKM-GBL-S, GF-S, GLT-S, GBLT-S, GFLT-S
 - Press Cure: 10 min/177°C Post Cure: 2 h/232°C in air

Following post cure, test specimens for all physical property and aging tests were die-cut from the molded slabs cut into standard dumbbells and tested per ASTM methods.

Formulation	Standard	Conventional Specialty	New APA Specialty
FKM- A601C	100		
FKM- GF, GBL-200, GFLT, GLT FKM- GF-S, GBL-S, GLT-S, GBLT-S, GFLT-S		100	100
High Activity Magnesium Oxide	3		
Calcium Hydroxide	6		
MT Carbon Black (N-990)	30	30	30
Zinc Oxide		3	3
VPA #2	0.5	0.5	0.5
Triallylisocyanurate		3	3
2,5-Dimethyl-2,5-di (tert-butylperoxy)- hexane, 45%		3	3
<i>Note: FKM-A601C contains bisphenol curative</i>			

Table 2. Compound formulations evaluated

A series of extended agings was conducted in gas turbine lubricant to ascertain elastomer compatibility. The lubricants selected represent a sampling of the synthetic aircraft engine lubricants commercially available today. They are shown in Table 3.

Designation	Description	Type
MJO II	Mobil Jet Oil II	Type II - MIL-PRF-23699 - STD
BPTO 2380	Air BP Turbine Oil 2380	Type II - MIL-PRF-23699 - STD
BPTO 2197	Air BP Turbine Oil 2197	Type II - MIL-PRF-23699 - HTS
MJO 291	Mobil Jet Oil 291	Type II - MIL-PRF-23699 - HTS

Table 3. Jet oils evaluated

Reference Fluids: Testing is often conducted with standard reference fluids in an effort to minimize variability between laboratories. One of the legacy aerospace reference fluids still used to characterize elastomer compatibility to di-ester (polyol) fluids is specified by AMS 3021. Historically, this fluid was produced by the Stauffer Chemical Co. and was referred to as “Stauffer Blend 7700”. Today, it is simply known as “Blend 7700” and is available from Akzo Nobel.

Development of Turbine Reference Oils for Elastomer Evaluation: In the late 1980’s lubricant engineers at Wright-Patterson Air Force Base developed a reference fluid based on trimethylol propane triheptanoate. This polyol fluid has typically been used to evaluate the ability of elastomeric and other polymeric materials to conform to designated requirements after contact with, or immersion in, Mil-L-7808 and similar lubricants at specified time and temperature, as required by the material specification, and its use is limited to such applications. This fluid is not intended for operational use in gas turbine engines. It is available directly from SAE as ARM-200 Fluid and is defined by AMS 3023.

As gas turbine engine technology has advanced, lubricants having greater thermal capability have become more widely used. In response to this industry need, Air BP recently developed a reference oil designed for compatibility testing with elastomeric

materials. The need for this new reference oil is driven by the growing popularity and use of more oxidatively stable aviation turbo oils. High Thermal Stability (HTS) oils tend to be more aggressive towards some of the more commonly used aviation grade elastomers. The current reference fluid (ARM-200) is no longer representative of current industry requirements. A new oil, Reference Oil 300, has been formulated to be representative of HTS-type jet oils. It became available October 2002 and may be purchased directly from Air BP.

Test Protocols - Standard Property and Aging Tests

The elastomers were characterized using the test methods described below:

- Cure response measured using a Moving Die Rheometer (ASTM D 6204-01)
- Mooney Scorch measured using a Mooney Viscometer (ASTM D 1646-90)
- Stress-strain properties measured according to ASTM D 412-98a
- Tear strength measured according to ASTM D 624-00, Die B
- Hardness measured according to ASTM D 2240-02
- Compression set properties according to ASTM D 395-01
- Temperature retraction tests (TR-10) according to ASTM D 1329-88
- Glass Transition (T_g) measurements made with a TA Instruments Modulated Differential Scanning Calorimeter, Model 2920, run in sub-ambient mode at a scanning rate of $5^\circ\text{C}/\text{min}$
- Fluid immersion according to ASTM D 471-98
- Heat aging tests according to ASTM D 573-99
- Compression Stress Relaxation measurements according to ASTM D 6147-97 using an H.W. Wallace C11 Mk III relaxometer and C11/9 oil evaluation jigs designed to apply a constant compressive strain to an O-ring test specimen (AS 568-113 size and tolerance)
- Molding Performance: To simulate the molding performance of the peroxide cured fluoroelastomers being evaluated; DuPont Dow's European Technical Center has developed a quantitative demolding test. The method evaluates the ease in which small O-rings can be removed from the simulated production O-ring mold shown in Figure 2.



Figure 2. O-ring mold used for demolding tests

The mold and test protocol used was described in a Rubber Division/ACS paper [12]. The mold consists of two sets of O-ring cavities of 60 parts each. The O-ring size and cross-section is similar to a fuel injector O-ring: a thick cross-section with a small internal diameter. This geometry produces a large surface area, which is advantageous for evaluating the mold-sticking phenomenon. Test compound is extruded using a Barwell preformer with a barrel temperature of 90°C operating at a sufficient pressure to ensure a smooth extrudate surface finish. The cord is then loaded into the mold and usually cured for 90 to 120 seconds at 177°C, and demolded with a controlled, light stream of air only. The mold is self-deflashing so that demolding of the O-rings is effected entirely by the air stream with no assistance from any flash that might be present.

The following procedure was used to evaluate demolding performance:

1. Each cavity was cleaned with a small rotary brass wire brush
 2. The total flat surface was cleaned with a brass wire brush
 3. The mold surface, including cavities, was washed with solvent (acetone was used)
 4. The mold was assembled in the press and heated to desired molding temperature
 5. Semi-permanent mold release agent was applied to both upper and lower mold surfaces
 6. The mold was loaded with extruded cord using a loading frame to position the cord directly over the cavities to ensure equal flow and cure time for each cavity (experimental material on one side and control material on the other)
 7. The mold was closed and material cured for the selected time
 8. O-rings were demolded using only air pressure; those, which remained stuck to the mold, were counted.
 9. Steps 6 through 8 were repeated until fifty (50) molding cycles were complete (50 molding cycles was previously determined to be sufficient to distinguish demolding performance between materials)
- Low temperature sealing performance was measured using a special test rig and protocol developed by DuPont Dow Elastomers. No established ASTM test method yet exists. This test rig has been previously described [13] and has been routinely used to screen the low temperature functionality of elastomeric sealing materials. The apparatus consists of a stainless steel block and plugs as shown in Figure 3, along with associated fasteners, valves, fittings and hoses. This apparatus was adapted, with major modifications, from SAE Aerospace Material Specification 7273B. The gland design conforms to SAE Aerospace Recommended Practices 1231 and 1232A. A radial squeeze of 19% is provided within three separate cells of the test block. The polished surface finishes employed are of industry standard (0.10µm / 4µin). Apparatus cooling is provided by a low temperature environmental chamber capable of achieving -70°C.

The test procedure consists of selecting three (3) AS568-214 O-rings of excellent visual surface quality being lightly lubricated with a low temperature Krytox[®] perfluorinated polyether grease prior to installation in the test plug gland. The plug is fastened into the block with socket head screws until finger

tight. A spring-energized Teflon[®] PTFE face seal provides secondary backup directly above the leak port. The apparatus is internally pressurized with nitrogen gas to 1.38 MPa (200 psi) and properly placed into the environmental chamber. The apparatus is then cycled down to -70°C at ~0.5°C per minute until leakage is detected via linear mass flow meters. The temperature is monitored continuously by RTD platinum thermocouples. The test reports the average temperature of the three cells at 5 Standard Cubic Centimeter per Minute (SCCM) leakage. All data is captured via a data acquisition system. The rig then auto-cycles back to room temperature and the procedure is repeated with another set of O-rings. The time to run the test varies, depending upon the glass transition temperature (T_g) of the material. With FKM polymers, 3-4 hours is typical.

Note: Krytox[®] performance lubricates and Teflon[®] fluorocarbon resins are registered trademarks of the DuPont Co.

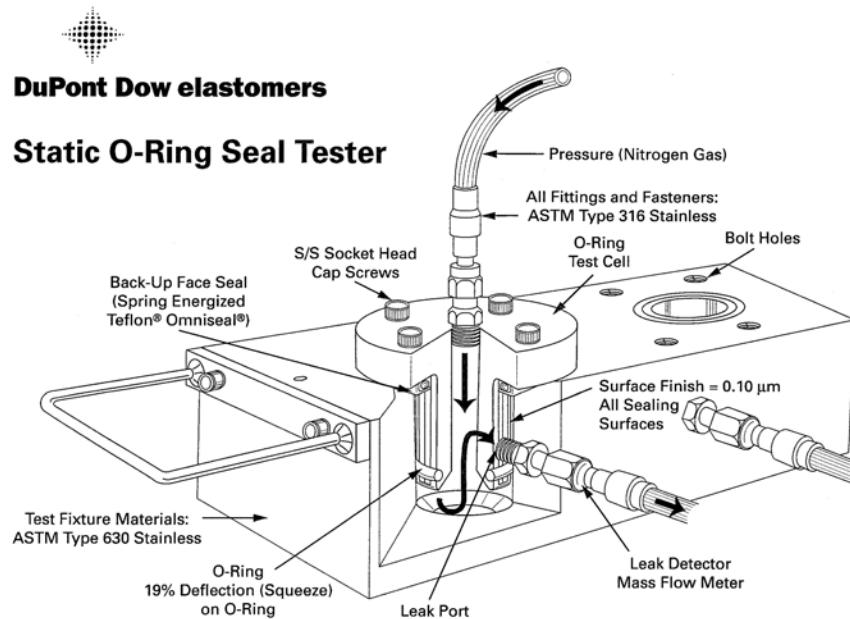


Figure 3. Low temperature test rig - Static O-ring Seal Tester

RESULTS and DISCUSSION

The testing reported in this study focused on methods used to characterize processing behavior, as well as properties relative to sealing performance. These topics will be discussed separately.

PROCESSABILITY OF APA POLYMERS

Rheology

Measurements of cure response were done using a Moving Die Rheometer and the results are graphed for GFLT-S (VTX-8550) in Figure 4 and compiled in Table 4.

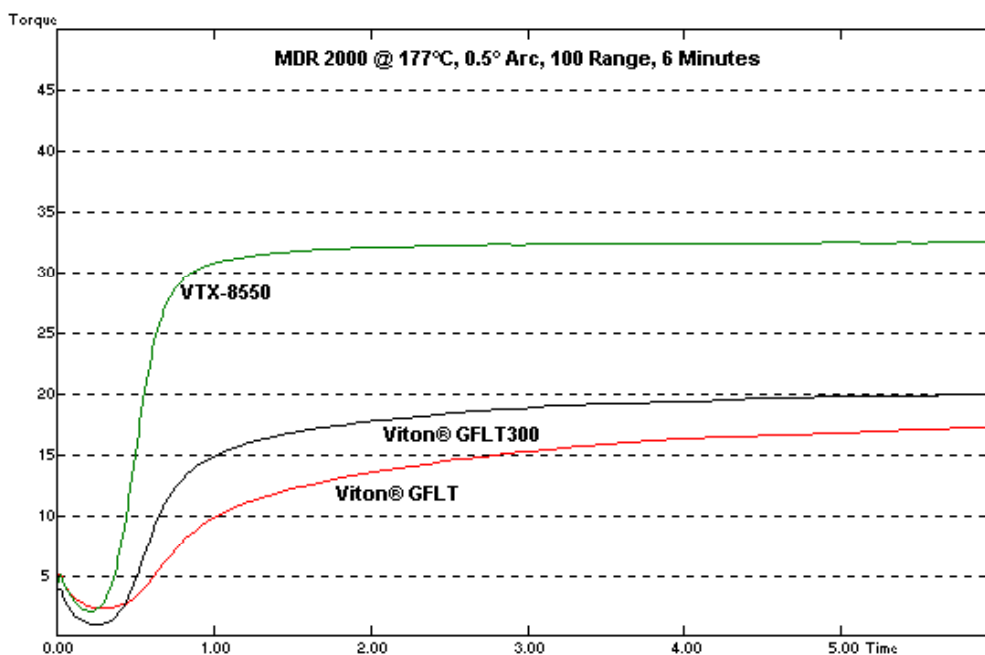


Figure 4. MDR cure curves for GFLT, GFLT300 and GFLT-S

As illustrated in Figure 4, there is dramatic difference in cure rate and cure state with polymers based on APA technology. The higher torque and faster cure rate clearly shows the more efficient cure response of GFLT-S (VTX-8550) when compared to older products like GFLT and GFLT300

		GFLT	GFLT-S
MDR2000 @ 177°C, 0.5° Arc			(VTX-8550)
ML	[dNm]	2.52	2.09
MH	[dNm]	17.57	31.66
ts1	[min]	0.49	0.31
ts2	[min]	0.57	0.35
T'50	[min]	1.01	0.53
T'90	[min]	3.78	0.8
T'95	[min]	5.03	0.98

Table 4. MDR cure data for GFLT and GFLT-S

Demolding Performance

In the O-ring demolding test, GLT-S (VTR-8500) was evaluated and compared to commercial GLT. Likewise, GBL-S (VTR-8650) was evaluated and compared to commercial GBL. The demolding evaluation data are shown below in Figure 5. The results are shown as a percentage of O-rings that were not removable by air pressure alone. The O-rings that stuck to the mold were not necessarily visually defective parts; merely they simply could not be removed using the air stream.

These demolding test results clearly show that peroxide cured GLT-S and GBL-S have a much lower propensity for mold sticking when compared to the conventional peroxide cured GLT and GBL products.

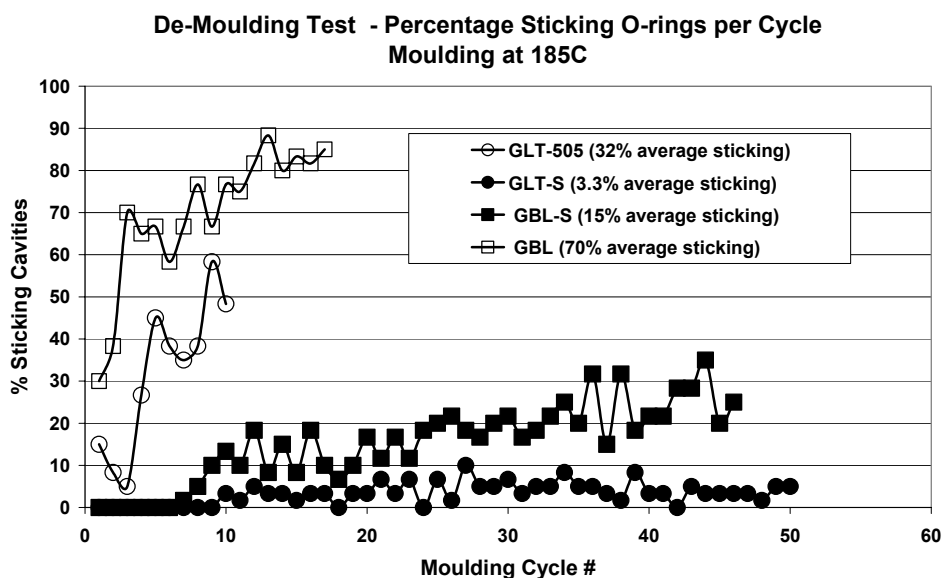


Figure 5. O-ring demolding results of APA vs. Old Technology products

PHYSICAL AND END USE PROPERTIES

The testing performed employed methods typically used to characterize elastomeric sealing performance. Hardness changes, volume swell, retention of stress-strain properties (tensile strength and elongation), compression set, compressive stress relaxation, and low temperature tests were performed.

Heat Aging Resistance

The heat resistance of the new polymers is similar to that of the older technology grades as indicated in Tables 5 and 6 that provides properties after hot air aging at 200°C for 1008 hours. This excellent heat resistance is largely due to the new curing chemistry - using APA technology it has been possible to achieve the dramatic processing improvements already described, without sacrificing the heat resistance for which FKM is so well known.

		GBL-S			GF-S	
		A601C	GBL	VTR-8650	GF	VTR-8600
Hot Air / 1008 hour / 200°C						
Hardness	[Shore A]	77	75	74	76	77
Hardness	[Pts. Change]	1	4	5	2	3
M10	[MPa]	1.2	1.0	1.0	1.1	1.2
M10	[% Change]	15%	14%	23%	14%	26%
M25	[MPa]	2.2	1.7	1.6	1.9	1.9
M25	[% Change]	15%	15%	21%	14%	20%
M50	[MPa]	3.9	2.9	2.4	3.8	3.1
M50	[% Change]	19%	31%	28%	36%	31%
M100	[MPa]	8.4	7.0	4.7	11.1	6.8
M100	[% Change]	20%	55%	39%	77%	36%
Tb	[MPa]	15.5	19.9	19.9	23.1	20.5
Tb	[% Change]	2%	12%	10%	27%	10%
Eb	[%]	185	207	299	184	275
Eb	[% Change]	-5%	-19%	-9%	-29%	-3%

Table 5. Heat Aging – 1008 hours @ 200°C - HFP-containing FKMs

		GLT-S		GFLT-S		GBLT-S	
		GLT	VTR-8500	GFLT	VTR-8550	VTR-8525	
Hot Air / 1008 hour / 200°C							
Hardness	[Shore A]	73	73	75	75	75	
Hardness	[Pts. Change]	5	5	5	4	5	
M10	[MPa]	0.9	0.9	1.0	1.0	1.0	
M10	[% Change]	23%	28%	20%	23%	22%	
M25	[MPa]	1.5	1.6	1.8	1.6	1.7	
M25	[% Change]	23%	25%	27%	19%	17%	
M50	[MPa]	2.7	2.3	3.7	2.6	2.5	
M50	[% Change]	37%	34%	58%	28%	22%	
M100	[MPa]	7.9	4.7	12.2	6.1	5.2	
M100	[% Change]	47%	42%	90%	30%	26%	
Tb	[MPa]	19.1	17.6	18.1	16.7	15.9	
Tb	[% Change]	12%	12%	9%	9%	7%	
Eb	[%]	175	275	133	229	228	
Eb	[% Change]	-17%	-7%	-32%	8%	-2%	

Table 6. Heat Aging – 1008 hours @ 200°C - PMVE-containing FKMs

Retained Elongation: Retention of elongation at break after aging is a valid measure of the resistance of a fluoroelastomer to property degradation (14). Elongation at break, and the changes which occur to this property during end-use service, are of particular importance to the functionality of static and dynamic seals. Therefore, the elongation retention after aging in the respective turbine lubricants will be reviewed at length.

Due to the extensive amount of data developed during this study, graphing will be by elastomer type and/or composition (HFP-containing vs. PMVE-containing), using tandem charts for each property and test condition, in an effort to enhance ease in viewing the graphics.

Volume Swell: Historically, volume swell, as performed in the laboratory, has been used as an indicator of an elastomer's chemical resistance to a specific fluid. Although it should not be used as the sole criterion for material selection, it is of value in predicting seal compatibility. The amount of volume swell in a given lubricant can affect sealing performance, as it relates to available O-ring groove volume, or abrasion and wear of dynamic sealing devices like radial lip seals.

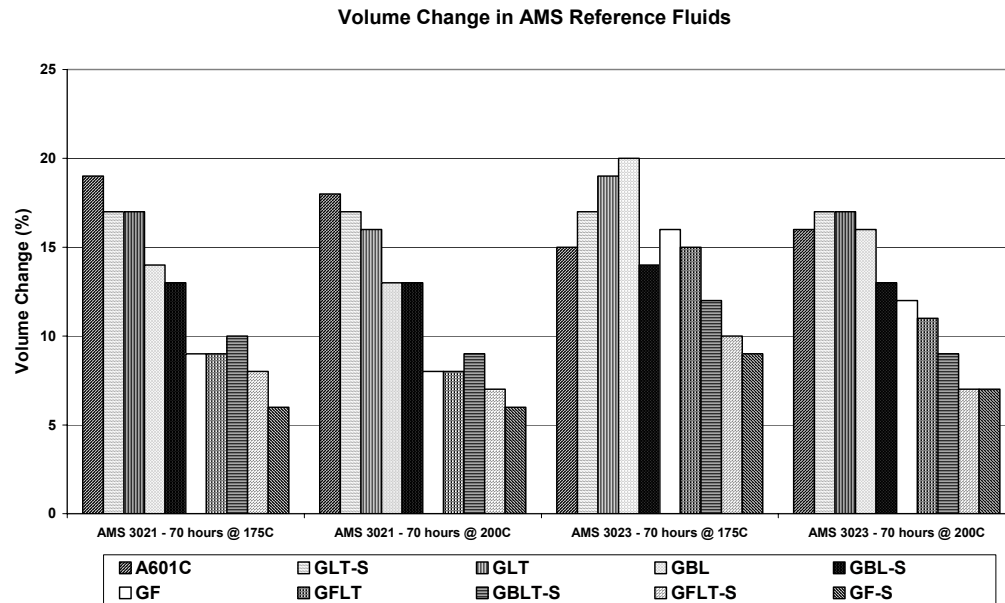


Figure 6. Volume change in AMS reference fluids

Figure 6 shows the volume swell in AMS 3021 and AMS 3023 reference fluids after 70 hour aging at 175°C and 200°C. As is frequently observed with such testing, the degree of swell is directly proportional to the fluorine weight percent contained within the polymer. Here FKM-GF and FKM-GF-S (70 wt % fluorine) and FKM-GFLT and FKM-GFLT-S (67 wt % fluorine) display the lowest swells in these fluids.

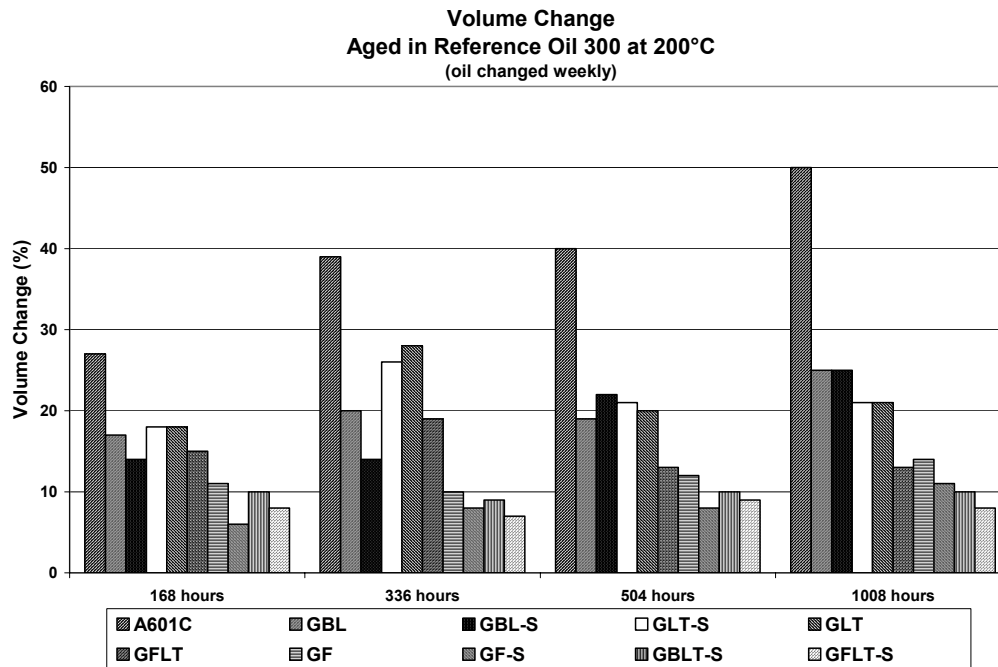


Figure 7. Volume change in Reference Oil 300 after 1008 hours at 200°C

Figure 7 shows the swell values for each FKM when immersed in the new Ref. Oil 300 through 1008 hours at 200°C. Here FKM-A601C displayed the highest swell (50%), while FKM-GF-S and FKM-GFLT-S showed the lowest swell values. Unquestionably, the new reference oil is significantly more aggressive to FKM, especially those polymers containing both VF2 and HFP.

Hardness Change: Typically, one of the affects of fluid swell in elastomers is a corresponding loss of hardness (softening). This generally holds true for the FKM materials reported in this study. Figures 8 show a strong relationship between hardness loss and the volume swell values shown in the preceding figure. With few exceptions, all of the materials evaluated lose hardness in these oils. The hardness loss can be appreciable. For example, FKM-A601C softened 25 points after 1008 hours immersion in Reference Oil 300 at 200°C. The best retention of hardness was seen with the PMVE-containing FKMs. Even after six weeks, all were within +/-5 points of their original hardness.

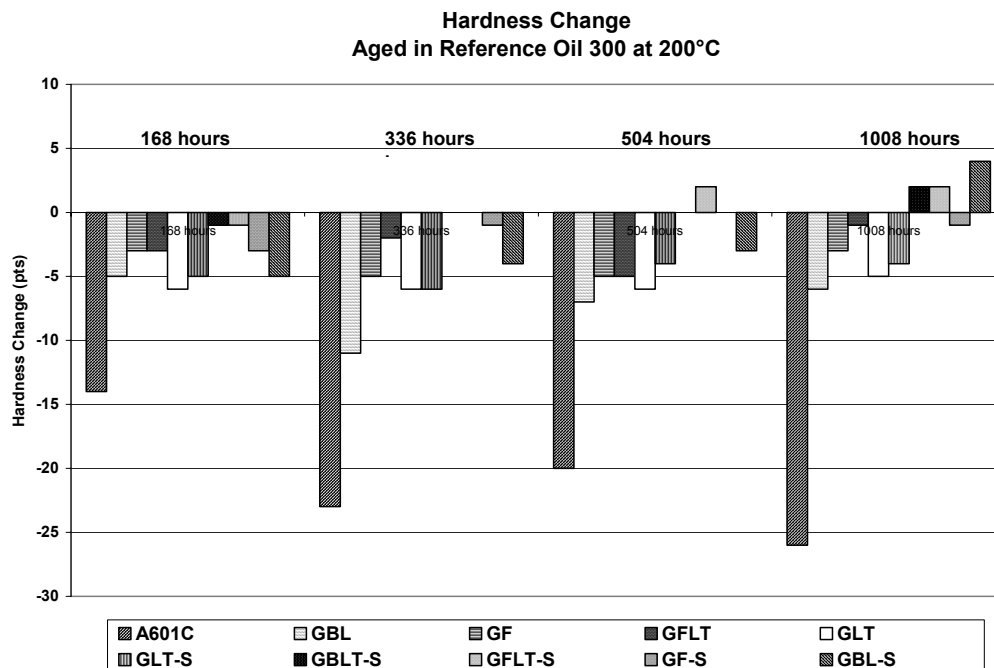


Figure 8. Hardness change after 1008 hours at 200°C in Reference Oil 300

Elongation Retention: Retention of elongation at break after aging is a valid measure of the resistance of a fluoroelastomer to property degradation [14]. Elongation at break, and the change of this property during end-use service, are of particular importance to the function of static and dynamic seals. Therefore, the elongation retention after aging in the respective turbine lubricants will be reviewed at length. Due to the large amount of data developed during this study, graphing will be by elastomer type and/or composition, using tandem charts for each property and test condition, in an effort to enhance the ease in viewing the graphics.

Figure 9 shows the significant loss of elongation with the HFP-containing fluoroelastomers after immersion in Ref. Oil 300 at 200°C. Elongation loss is dramatic and progresses with time, ultimately manifesting itself as embrittlement of the test specimen. FKM-A601C showed less than 5% retained elongation after 1008 hours.

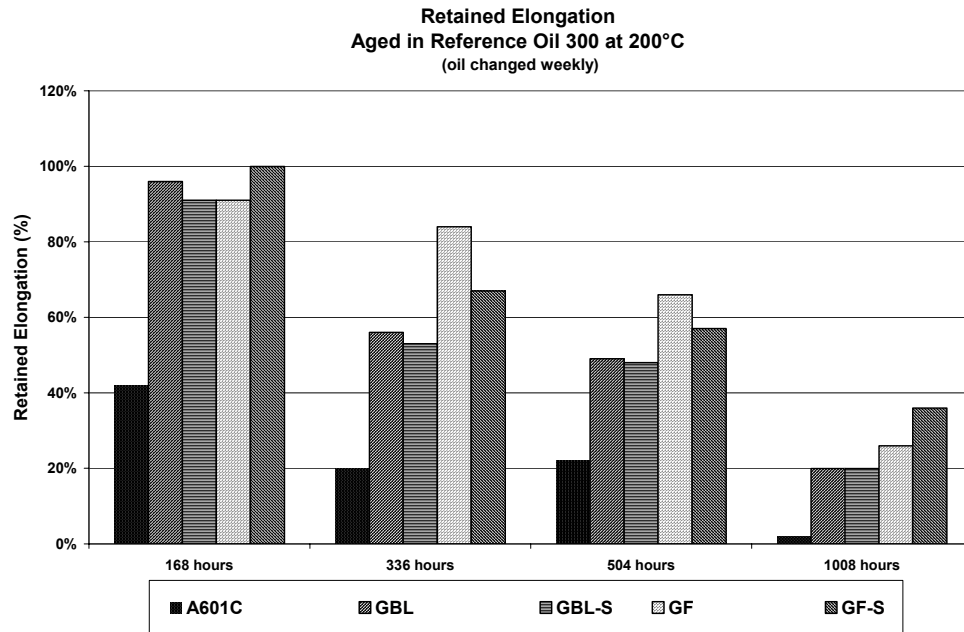


Figure 9. Elongation retention after 1008 hours at 200°C in Reference Oil 300

The outstanding resistance of PMVE-containing FKM to Ref. Oil 300 can be seen in Figure 10. All materials evaluated retained 75-80% of their original elongation.

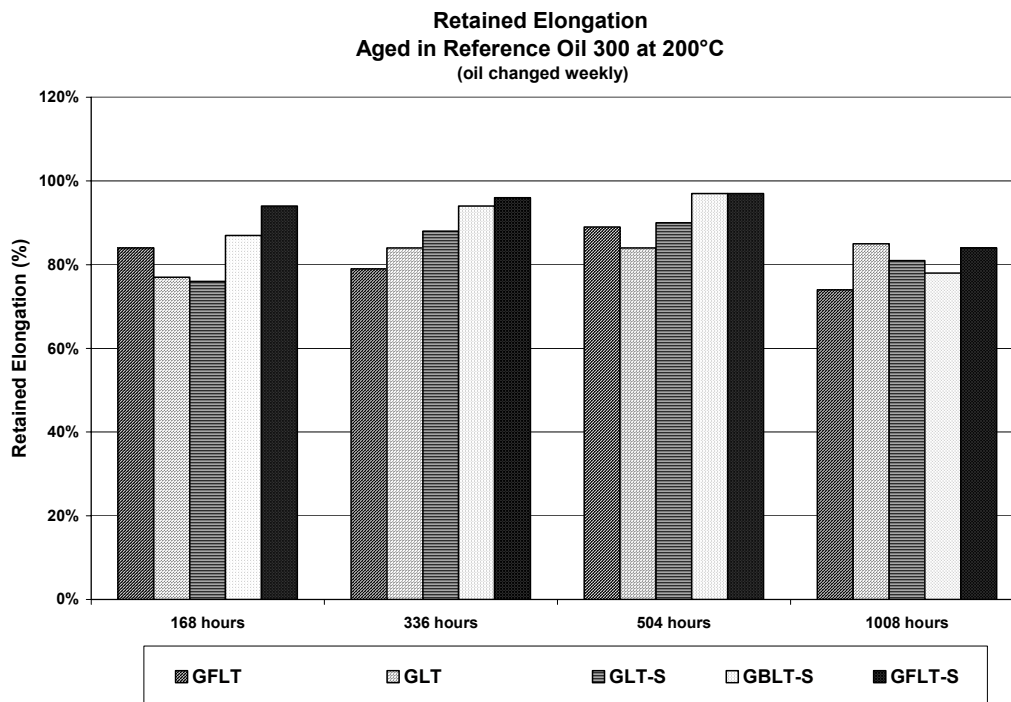


Figure 10. Elongation retention after 1008 hours at 200°C in Reference Oil 300

Jet Oil Compatibility Testing

As previously mentioned, gas turbine engine design is pushing fluoroelastomers to their limits. This design progression encompasses the relentless drive towards engines of higher thrust, lower weight, and greater fuel efficiency, with accompanying reductions in noise and emissions, as well as better reliability, operating safety and longer time on wing. These trends converge to push engine thermodynamics to their limits, invariably culminating in higher operating temperatures. As a result, engine builders have adopted advanced lubricants with higher thermo-oxidative stability (HTS), in order to extend engine life. Aircraft engine lubricants have had to maintain pace with this drive towards higher operating temperatures, while still meeting stringent demands for viscosity stability, anti-coking requirements, and regulatory and environmental compliance. In order for lube oils to be stable at higher temperatures, different and more aggressive additives are often used in conjunction with sophisticated packages of surfactants and amine-based stabilizers. It is primarily these additive packages that challenge the traditional fluoroelastomers to maintain long-life, leak-free sealing performance.

The effect of the newer lube oils, particularly those with amine stabilizers, on FKM-based seals was not generally appreciated until several serious failures were noted, particularly when soak-back conditions prevailed [15]. Service experience with auxiliary power units, starter motors, oil filters and other components, involving O-rings and other seal shapes, indicate that there are FKM compatibility problems with the latest generation of HTS lubricants. To meet this need, fluoroelastomers with better base resistance are being adopted. The material of choice, at least for the time being, is FKM-GLT. It is being specified via AMS-R-83485. Appendix II reviews the specification requirements and the properties obtained with FKM-GLT and FKM-GLT-S.

The Effect of Additives on Fluoroelastomers: Engine seals are exposed to hot oils containing aggressive amine-based stabilizers. It is primarily the amine additives that challenge traditional fluorohydrocarbon elastomers containing vinylidene fluoride (VF₂) to maintain long life, leak-free sealing performance. When VF₂-containing fluoroelastomers come in contact with amine-based additives (containing nitrogenous organic compounds), they are attacked and undergo dehydrofluorination to form unsaturation (double bonds) in the polymer backbone. These unsaturated sites are subject to crosslinking by chemical and thermal reactions. The newly formed crosslinks, in addition to the crosslinks already present by conventional curing, impart an increased state of cure as evidenced by a reduction in elongation (elasticity). When attack is severe, the rubber embrittles and develops surface cracks. As the strength of the base is increased, the rate of attack is increased resulting in the generation of additional unsaturation in the rubber backbone and further loss of elongation. An illustration of a proposed mechanism is shown in Figure 11.

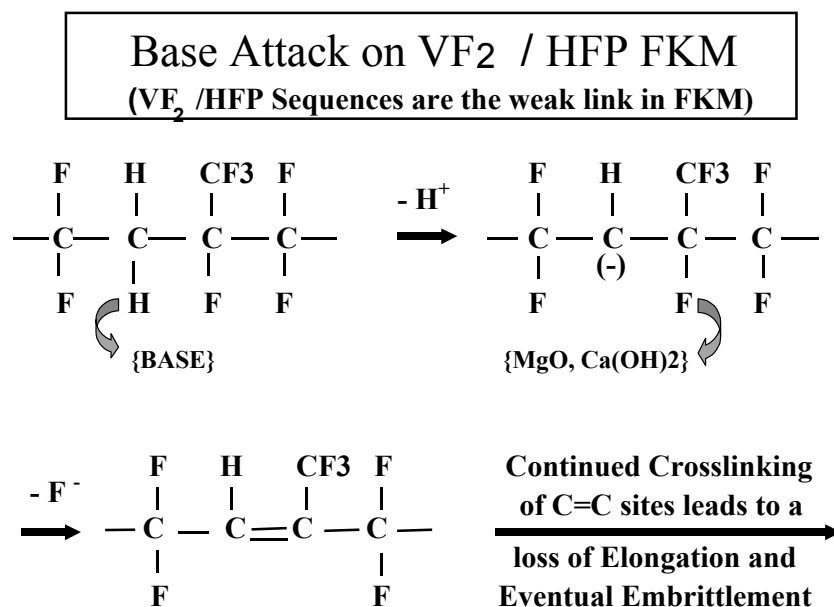


Figure 11. A proposed mechanism of base attack on VF₂ / HFP FKM

As mentioned earlier, retention of elongation is a valid, general measure of the resistance of a vulcanizate to degradation [16]. Figures 12-19 will examine fluoroelastomer resistance to elongation loss when immersed in the respective jet oils for 168, 336, 504 and 1008 hours duration at 200°C. Test results will be reported following the type of oil (Standard or HTS). Oils were changed weekly during testing. No aeration of the oil was attempted.

Elongation Retention in Type II Standard Oils: As mentioned previously, base attack on FKMs is dependent upon time, temperature and additive type. Figures 12 and 13 show fluoroelastomer aging after 1008 hours at 200°C in Mobil Jet Oil II. Note the progressive loss of elongation as a function of time. FKM-A601C, GF, GBL, GBL-S and GF-S exhibited elongation losses of over 60-80% in MJO II.

In contrast, although the PMVE-containing FKMs have similar (or lower) fluorine contents, they retain well over 70-85% of their original elongation. This is a result of the absence of HFP. This resistance to attack by jet oils will be a common theme observed throughout the balance of this study.

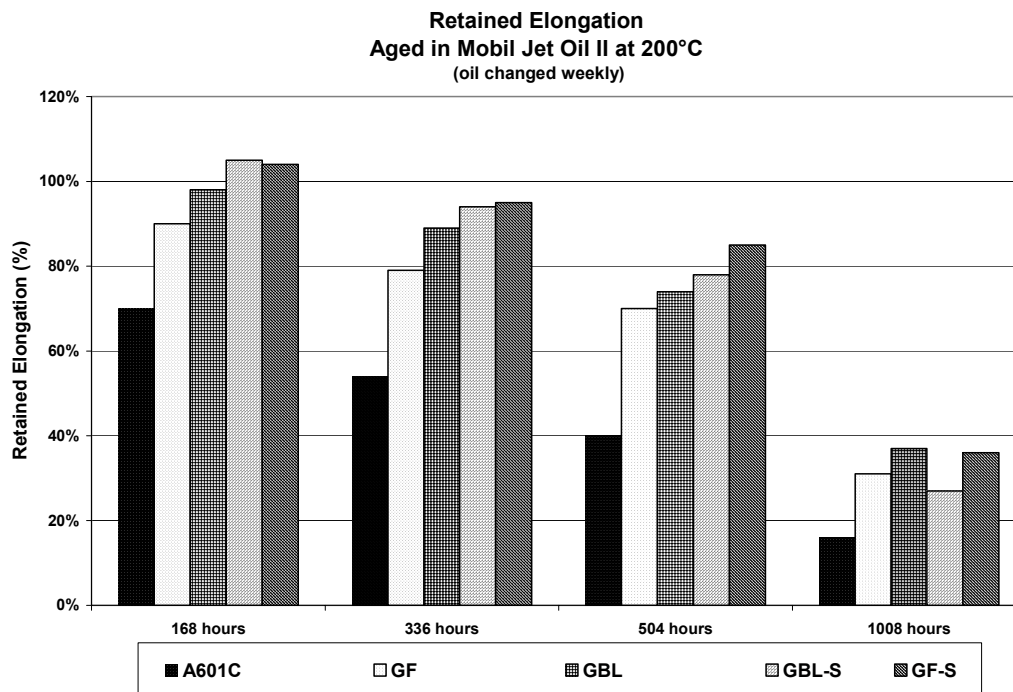


Figure 12. Elongation retention after 1008 hours at 200°C in Mobil Jet Oil II - HFP-containing FKMs

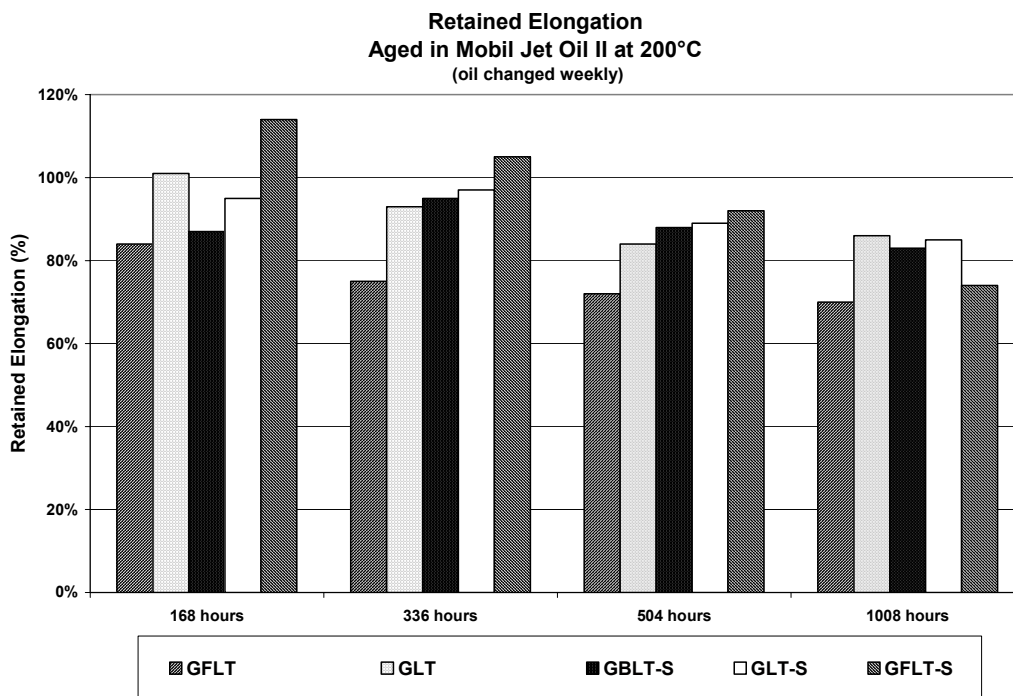


Figure 13. Elongation Retention after 1008 hours at 200°C in Mobil Jet Oil II - PMVE-containing FKMs

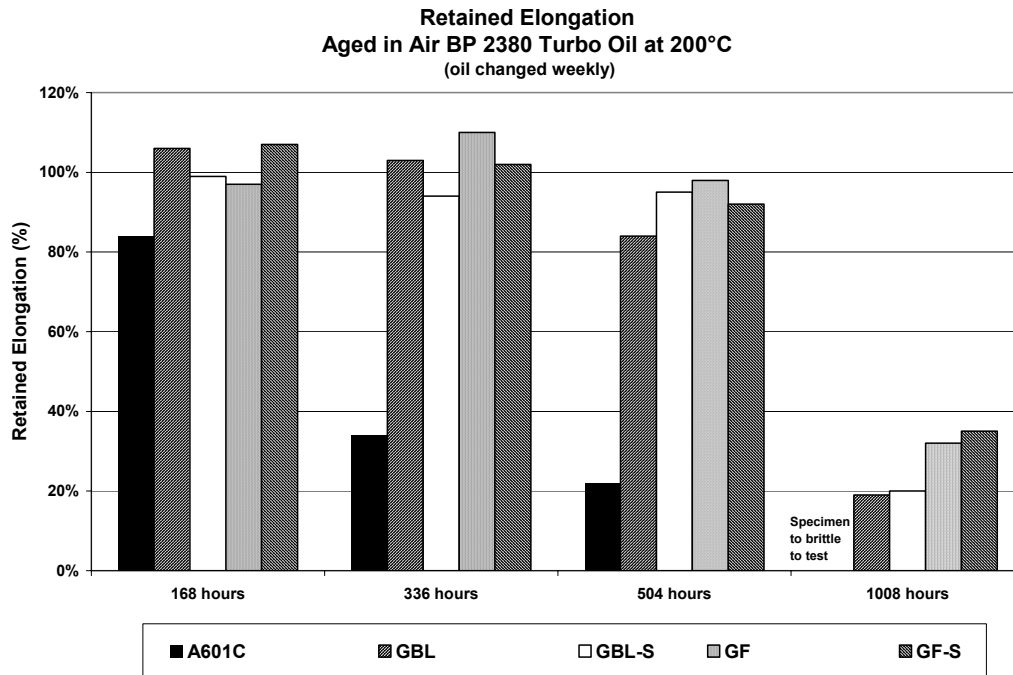


Figure 14. Elongation retention after 1008 hours at 200°C in BP Turbo Oil 2380

Figures 14 and 15 show retained elongation in BPTO 2380. Test results parallel those observed with MJO II, although the degree of retained elongation is less. FKM-A601C was too brittle to test after 1008 hours at 200°C. FKM-GLT, GFLT, GLT-S, GBLT-S and GFLT-S show good retention of elongation (80%) in this standard oil.

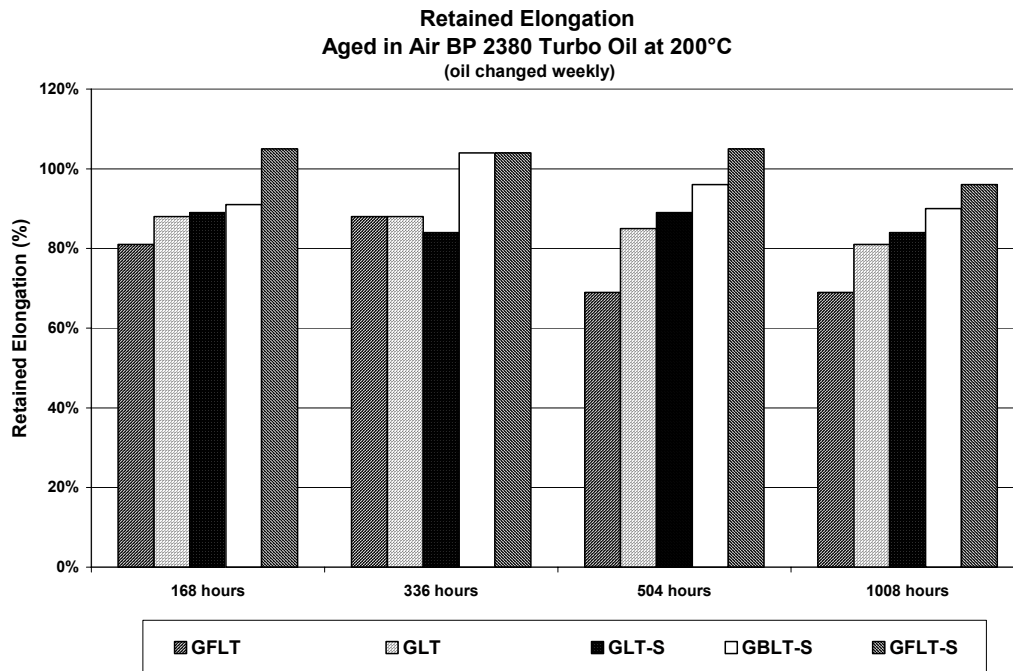


Figure 15. Elongation retention after 1008 hours at 200°C in BP Turbo Oil 2380

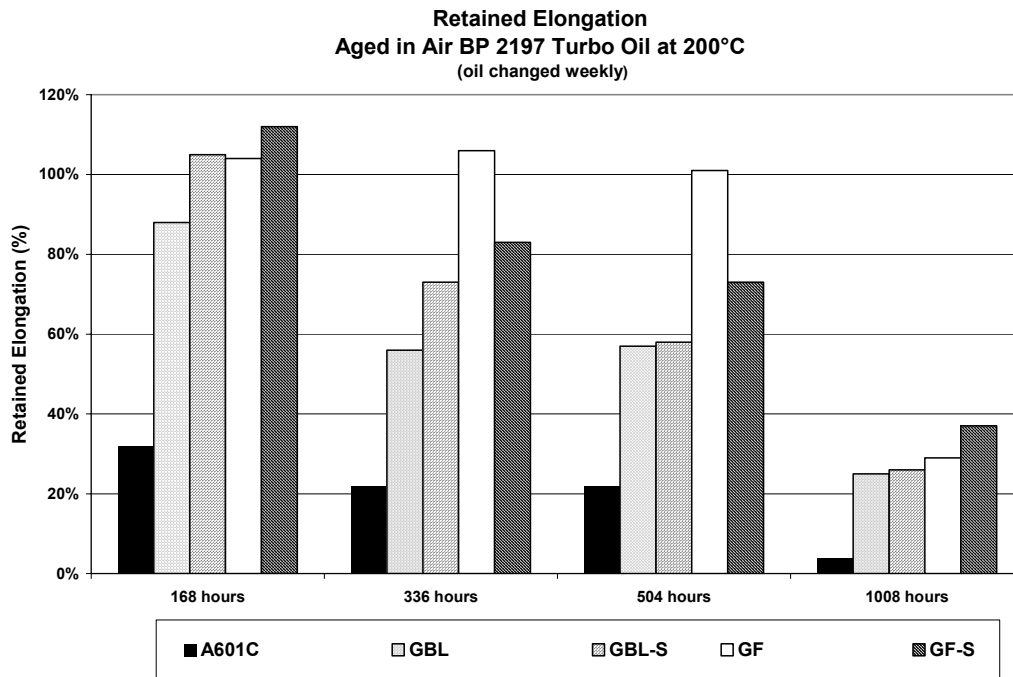


Figure 16. Elongation retention after 1008 hours at 200°C in BP Turbo Oil 2197

Figures 16 and 17 show retained elongation in BPTO 2197. Test results parallel those observed with MJO II and BPTO 2380, although this HTS oil is more aggressive toward the HFP-containing FKMs. FKM-GLT, GFLT, GLT-S, GBLT-S and GFLT-S show very good retention of elongation (75-80%).

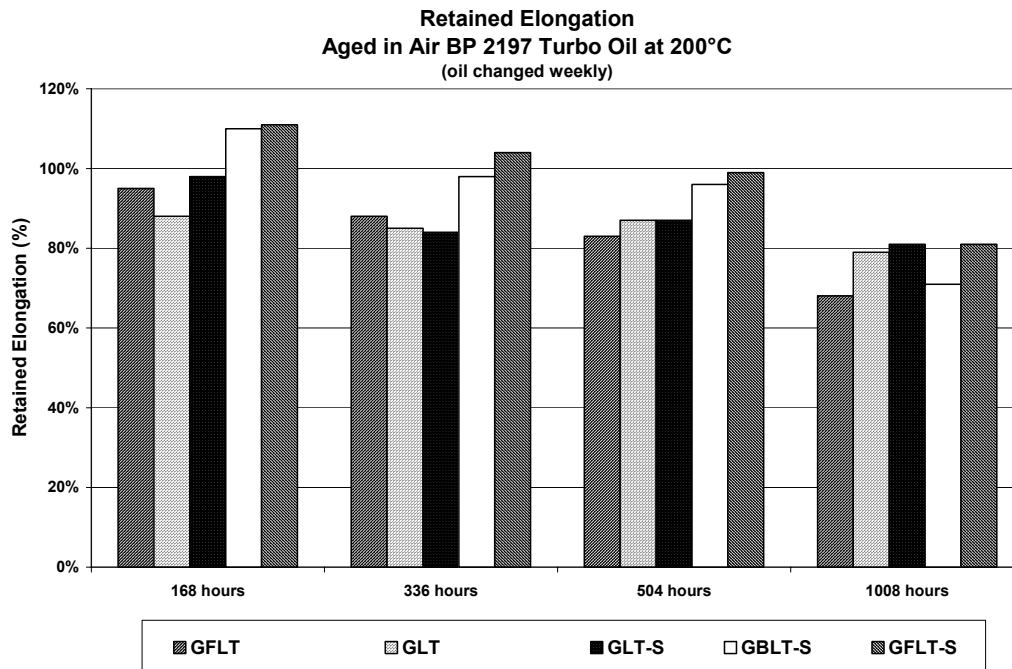


Figure 17. Elongation retention after 1008 hours at 200°C in BP Turbo Oil 2197

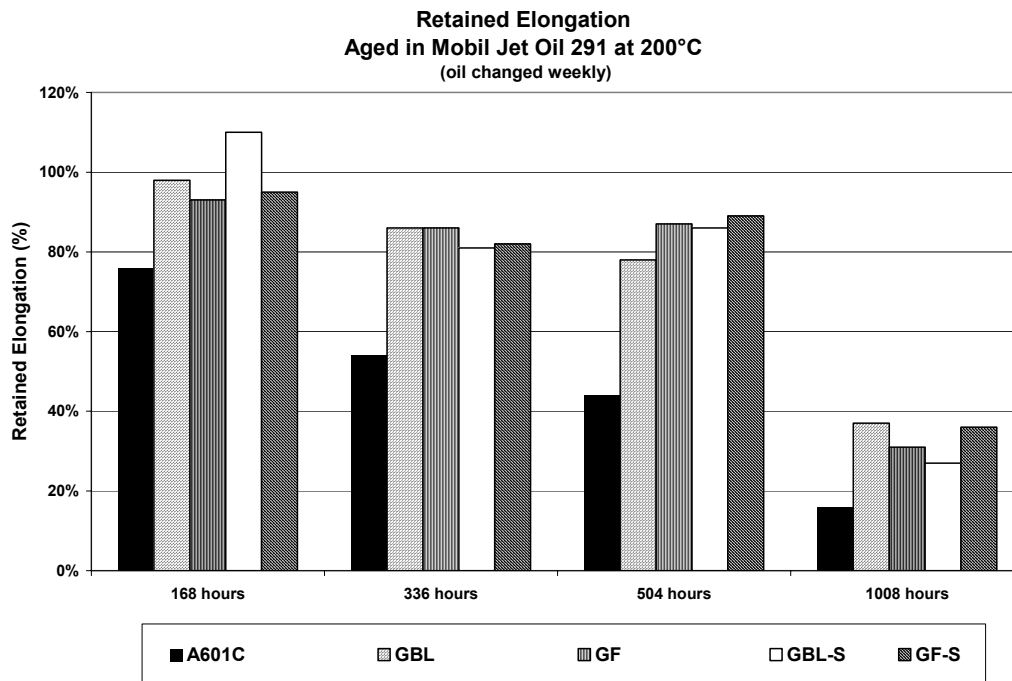


Figure 18. Elongation retention after 1008 hours at 200°C in Mobil Jet Oil 291

Figures 18 and 19 show retained elongation in MJO 291. Test results parallel those observed with BPTO 2197. This HTS oil is more not quite as aggressive toward the HFP-containing FKMs. Again we see FKM-GLT, GFLT, GLT-S, GBLT-S and GFLT-S display very good retention of elongation (75-80%).

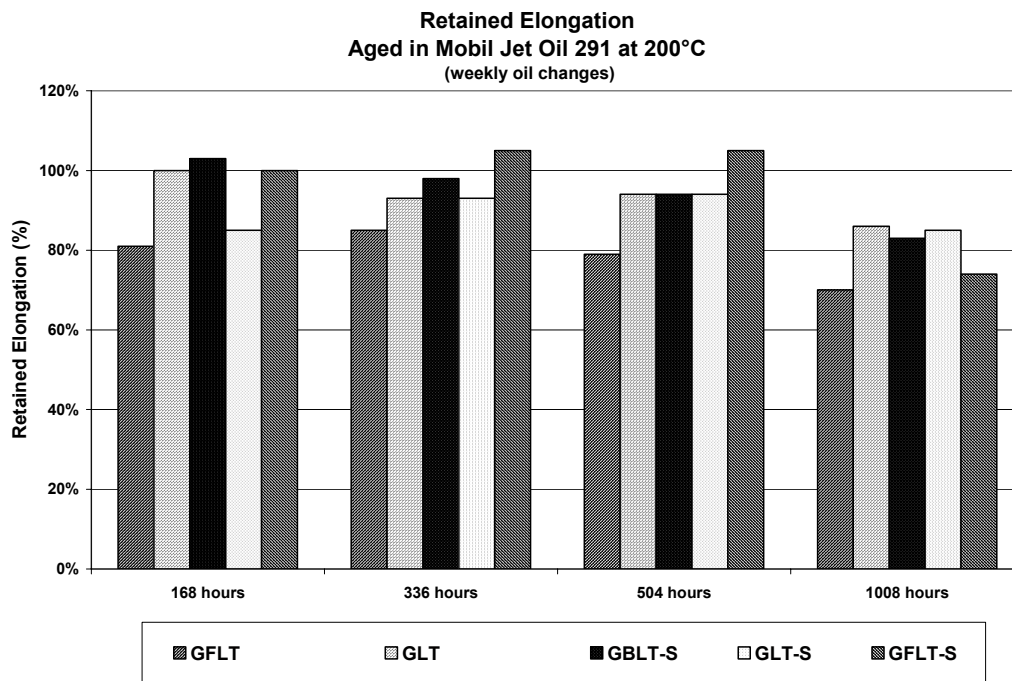


Figure 19. Elongation retention after 1008 hours at 200°C in Mobil Jet Oil 291

Sealing Under Compression

When elastomers are used as sealing devices, they are compressed or strained to some degree. When that strain is removed after a period of time, the material will not fully recover its original shape. Creep, stress relaxation and compression set are undesirable related phenomena, which occur in all elastomeric articles and reflect the inherent viscoelastic nature of the elastomer and the limited stability of crosslinks of its vulcanizate.

Creep is a time dependent increase in deformation under conditions of constant stress [17]. Continuous stress relaxation is decay in stress, as a function of time, under conditions of constant strain [17]. It is of great importance to sealing devices such as O-rings and gaskets. Both creep and stress relaxation are significant because they often play a role in the failure of rubber components. Set, often referred to as "permanent set", or irrecoverable creep, is the permanent deformation which remains when a material is released from the strain imposed and is measured in tension, or more commonly in compression [17].

Both stress relaxation and creep are the result of physical and chemical relaxation processes [18]. The physical process is due to the viscoelastic nature of rubber and usually decreases linearly with the logarithm of time. It is associated with reorientation of the elastomer molecular network under strain, with disengagement and rearrangement of chain entanglements. The chemical process is generally caused by chain scission or isomerization of crosslinks and usually occurs linearly as a function of linear time. The time dependence of the chemical process is a function of the order of the chemical reaction, the temperature and the imposed stress. Both processes occur simultaneously. Physical relaxation predominates at short times, while chemical effects are more significant at longer duration.

When an elastomeric component is subjected to a static load, the load will cause a progressive increase in deformation as a function of time. Bi-axial stress relaxation on compression in rubber consists of both physical creep and chemical creep (due to molecular chain breaking). When a constant strain is imposed on an elastomer, the force necessary to maintain that strain is not constant but decays exponentially with time from the initial maximum to an eventual equilibrium state [19]. This phenomenon of force decay is called stress relaxation and is of great importance in rubber sealing devices such as O-rings, packings and gaskets. Stress relaxation can be the dominant factor that limits the effective seal life.

Compression Set: In this method, the test specimen is compressed 25%, held under compression for a specified time at temperature, and subsequently released. Measurements are then made to determine how a material recovers from this deformation. Testing can be performed in gas (typically air) or immersed in a fluid. In this study compression set was run per ASTM D 395, Method B, on -214 O-rings.

Figures 20 and 21 show the compression set results obtained for the various fluoroelastomers when tested in 200°C air for 22, 70, 336, 504 and 1008 hours. The shorter duration testing has been routinely specified in AMS sealing documents. As the industry moves towards longer time on wing, longer term testing is becoming increasingly necessary. After two weeks (336 hours) at 200°C, significant changes are becoming evident between the materials. The thermal capabilities of the crosslink

network employed are becoming apparent. The best long-term (1000 hour) compression set properties was observed with FKM-A601C, which employs a thermally robust bisphenol cure system.

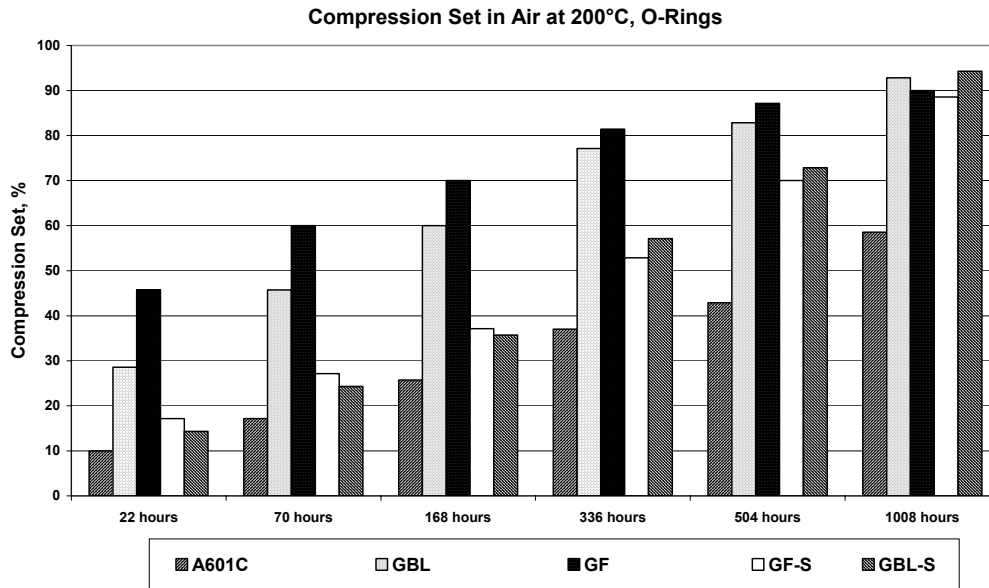


Figure 20. Compression set after 1008 hours at 200°C – HFP-containing FKM

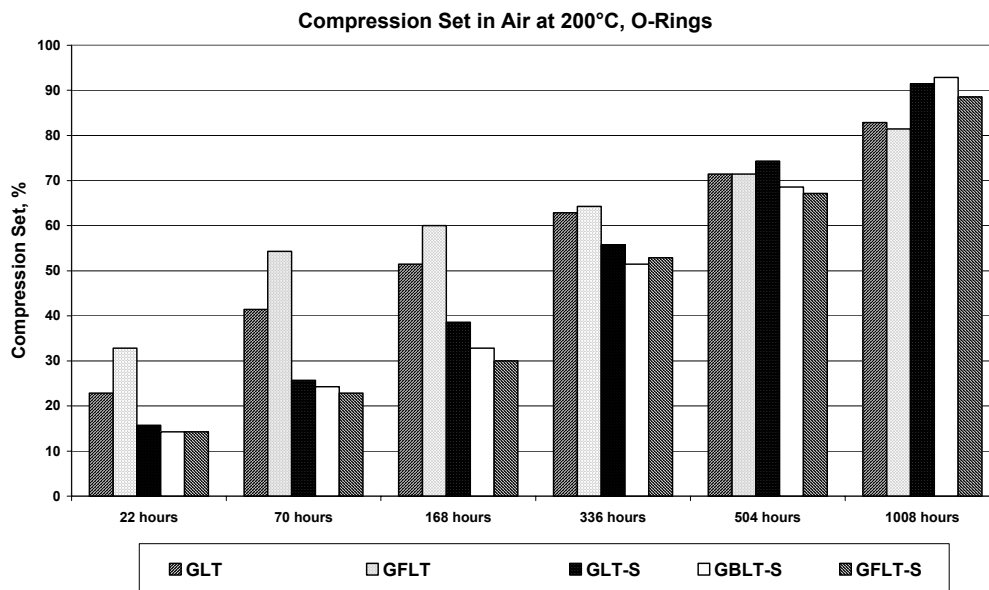


Figure 21. Compression set after 1008 hours at 200°C – PMVE-containing FKM

Stress Relaxation: The stress relaxation data reported in the paper was obtained using Shawbury-Wallace oil evaluation jigs and apparatus and tested per the ASTM D 6147 method. Other styles of apparatus have been recently documented [20] and each has

value, as well as limitations. What we seek is a useful and repeatable tool to help us predict long-term elastomeric sealing performance. Unquestionably, stress relaxation is certainly becoming a valued tool to the aerospace sealing community.

In our study we tested in Mobil Jet Oil 291 oil for 2016 hours (12 weeks) at 200°C. Figure 22 shows the percentage of compressive force retained as a function of time in MJO 291. Here we can see how the compressive force decays over time for all the fluoroelastomers. Well over 50% of the original sealing force is lost in the first 100 hours. Then the decay begins to moderate. FKM-GLT-S showed the best performance, retaining 36% compressive force. This was followed by FKM-GLT (15%) and A601C (11%).

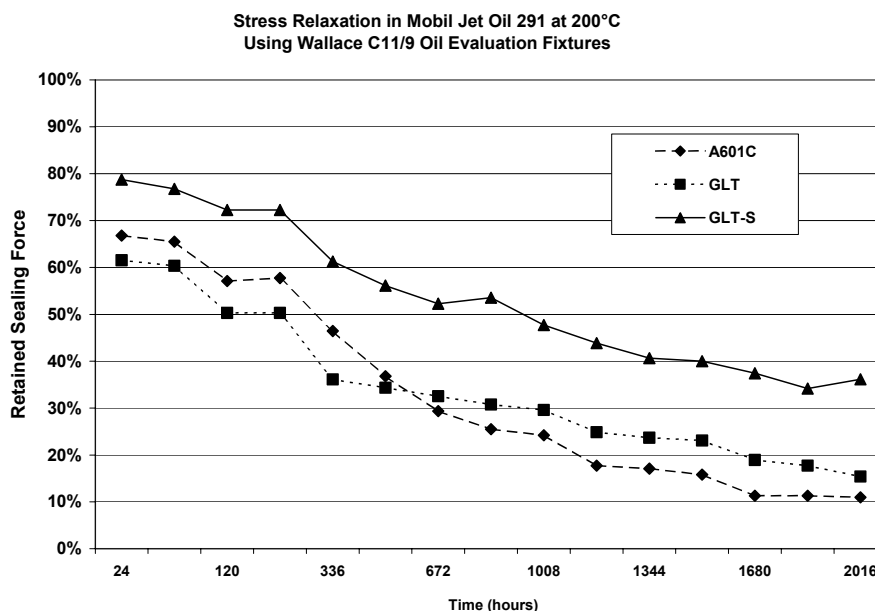


Figure 22. Sealing force retention of GLT-S, GLT and A601C after aging in MJO 291 jet oil for 2016 hours at 200°C

Discussion of Low Temperature Property Testing and Sealing

Low temperature flexibility is strongly influenced by the level of VF₂ in the polymer, even for those polymers in which PMVE is used to lower the glass transition temperature. The very specific combinations of main monomers which provide the excellent balance of fluid resistance and low temperature flexibility exhibited by compositions like FKM-GLT was determined in the mid-1970's.

Low Temperature Properties

At low temperatures the modulus of an elastomeric sealing element increases as the temperature decreases. As the sealing material approaches its glass transition temperature (T_g), hardness increases and it ultimately loses its ability to remain flexible [21]. Sealing force decays very rapidly. Perhaps nowhere was the reality of this intractable physical phenomenon more painfully evident than the tragedy that befell NASA's Mission 51-L in 1986 (the Challenger disaster).

Figure 23 shows the characteristics of the various materials tested under sub-ambient conditions. Here we compare the results obtained from several test methods characterizing low temperature properties.

FKM-A601C has a glass transition temperature of -17°C and a temperature retraction value of -16°C . This requirement (TR-10) is reflected in the major FKM aerospace seal documents presently in use (AMS 7276, AMS 7259, AMS 3216, AMS 3218). FKM-GBL has a T_g of -16°C . Note that GF has the poorest low temperature properties, the expected result of its low VF2 content. The best low temperature properties were observed with the PMVE-containing fluoroelastomers – FKM-GLT and GFLT, and the new APA-grades, FKM-GLT-S, GBLT-S and GFLT-S. This advantage is particularly evident in the static O-ring seal testing. MIL-R-83485 was written around the low temperature properties of FKM-GLT in 1976. Since then, it has undergone a word-for-word conversion to AMS-R-83485 and is presently being updated to include Reference Oil 300 aging and longer-term compression set test requirements.

Comparison with temperature retraction (TR-10) and glass transition (T_g) shows good correlation. From the static O-ring seal testing, we see about a -15°C correlation between low temperature sealing values and those obtained from TR-10 and T_g methods. The ability for O-rings to seal at low temperature correlates closely with glass transition and temperature retraction. More importantly, the ability for fluoroelastomer O-rings to seal at low temperature, as determined by this method, is predominately dependent upon polymer composition.

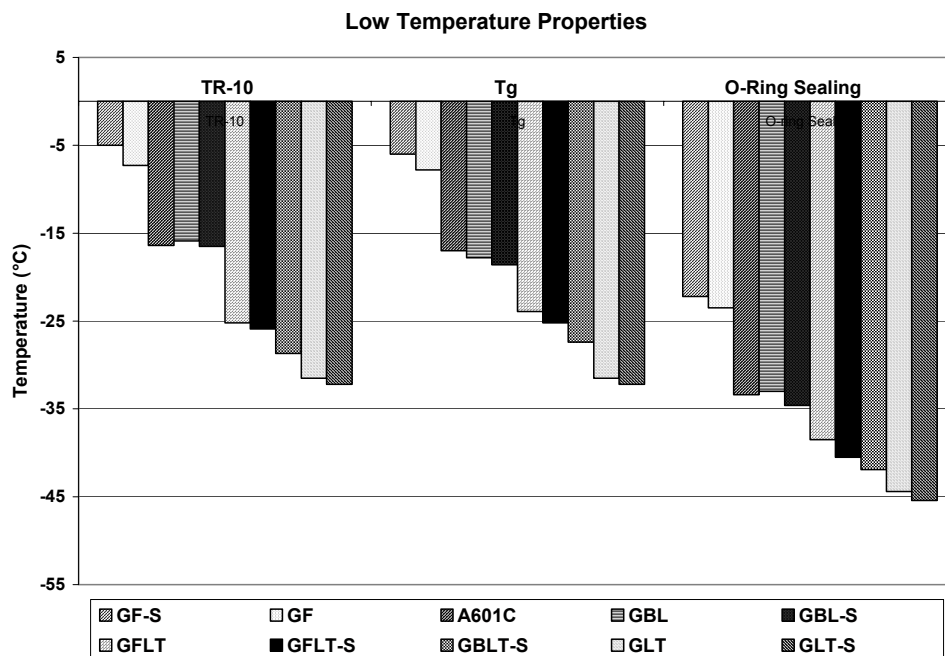


Figure 23. Comparison of low temperature results from various test methods

SUMMARY AND CONCLUSIONS

The objective of this paper was to review a new series of peroxide curable specialty fluoroelastomers that represent a considerable upgrade over the products traditionally available from DuPont Dow Elastomers.

A range of specialty elastomers has been reviewed to evaluate their compatibility with advanced gas turbine engine lubricants and reference fluids. Although conventional VF₂/HFP fluoroelastomers continue to be used in commercial and military engines, the specialty types of fluoroelastomers containing PMVE evaluated in this paper offer superior compatibility with the newer oils, especially the HTS-types. For this reason, materials like FKM-GLT and the newer PMVE-containing APA grade, FKM-GLT-S (VTR-8500), will find increasing adoption because of their superior balance of service temperature range, retained sealing force and property retention in advanced jet oils.

The processing benefits of APA technology were highlighted and several of the advantages of these new products have been illustrated with supporting data.

A brief summary of the principal advantages offered by the new APA polymers is:

- Faster cure response and a higher state of cure provides improved productivity through significantly reduced cure cycle times.
- A new curing system combined with redesigned polymer end groups and polymer architecture provides a step-change in mold release and reduction in the tendency for mold fouling. This will improve processing yields and give significant cost savings through reduced scrap and increased productivity.
- A further result of reduced mold fouling is that tooling will require less frequent cleaning, which will contribute to improved productivity and longer tool life.
- The general physical properties of new technology polymers are generally superior to the old technology grades.
- The compression set resistance of the new technology polymers is much improved and should provide better long term sealing performance. This is backed up with long-term stress relaxation data at high temperatures.
- The heat and fluid resistance of the new technology products is comparable to older grades and in some cases is substantially better.
- The new technology products have superior low temperature sealing ability.

Viton[®] fluoroelastomers made with APA technology are expected to deliver considerable benefits to aerospace part manufacturers and end users alike, providing improved processing, better part quality, higher manufacturing yields, and more durable service performance.

ACKNOWLEDGMENT

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Appendix I

Compounding Ingredient Designation	Ingredient Used	Supplier
FKM-A601C	Viton [®] A-601C	DuPont Dow Elastomers
FKM-GBL	Viton [®] GBL-200	DuPont Dow Elastomers
FKM-GF	Viton [®] GF	DuPont Dow Elastomers
FKM-GFLT	Viton [®] GFLT	DuPont Dow Elastomers
FKM-GLT	Viton [®] GLT	DuPont Dow Elastomers
FKM-GBL-S	Viton [®] VTR-8650	DuPont Dow Elastomers
FKM-GF-S	Viton [®] VTR-8600	DuPont Dow Elastomers
FKM-GLT-S	Viton [®] VTR-8500	DuPont Dow Elastomers
FKM-GBLT-S	Viton [®] VTR-8525	DuPont Dow Elastomers
FKM-GFLT-S	Viton [®] VTR-8550	DuPont Dow Elastomers
Carbon Black N-990	MT Black N-990	J. M. Huber Corp.
Calcium Hydroxide	Calcium Hydroxide HP	C. P. Hall
Austin Black	Austin Black 325	Harwick Standard Distribution
Zinc Oxide	Zinc Oxide	Zinc Corp. of America
High Activity Magnesium Oxide	Elastomag [®] 170	Morton International
2,5-Dimethyl-2,5-di (tert- butylperoxy)-hexane, 45%	Varox [®] DBPH-50	R.T. Vanderbilt Co.
Triallylisocyanurate	Diak [™] #7	DuPont Dow Elastomers
Viton [®] Process Aid #2	VPA #2	DuPont Dow Elastomers
Carnauba Wax	Carnauba Wax	F. B. Ross Co.

Appendix II

Evaluation of GLT vs. GLT-S N990 & N990 / Austin Black Compounds for AMS-R-83485 Specification

Polymer / filler	GLT / MT	GLT-S / MT	GLT / Austin	GLT-S / Austin
Viton GLT	100	-	100	-
Viton GLT-S	-	100	-	100
Zinc Oxide	3	3	-	3
Calcium Hydroxide	-	-	3	-
N-990	30	30	10	10
Austin Black 325	-	-	20	20
Carnauba wax	0.5	0.5	0.5	0.5
Diak #7	3	3	4	4
Luperox	3	3	4	3
101XL45				
Total	139.5	139.5	141.5	140.5

Mooney Scorch @ 121°C

	GLT	GLT-S	GLT	GLT-S
Minimum	87	55	91	61
2-pt rise (min)	16.1	15.1	8.6	8.3
5-pt rise (min)	18.7	16.3	10.1	9.6
10-pt rise (min)	20.5	17.1	11.1	10.5

MDR 2000 @ 177°C, 0.5°Arc, 6 Minute Clock

	GLT	GLT-S	GLT	GLT-S
M-L (dNm)	3.1	1.9	4.4	2.2
ts-2 (min)	0.6	0.4	0.6	0.4
t'50 (min)	1.0	0.6	1.0	0.6
t'90 (min)	3.1	1.0	3.0	1.1
t'95 (min)	4.0	1.5	3.9	1.7
M-H (dNm)	16.9	25.4	23.9	31.9

Physical Properties @ R.T. - Original (Cured 7' @ 177C – Post-cured @ 232°C as noted)

	16 hrs PC	4 hrs PC	16 hrs PC	4 hrs PC	AMS-R-83485
M-10 , MPa	0.77	0.82	0.93	0.83	
M-100 , MPa	6.1	3.7	10.5	4.9	
T-B , MPa	18.5	16.3	15.5	11.2	
(Tb, psi)	2678	2366	2242	1617	1600 min
E-B , %	192	310	158	304	120 min
Hardness, A, pts	71	71	75	72	75 +/- 5

Appendix II (cont.)

Polymer / filler =	GLT / MT	GLT-S / MT	GLT / Austin	GLT-S / Austin	AMS-R- 83485 <u>Spec Limits</u>
Compression Set , Method B , O-rings					
70 Hrs @ 23°C	14	9	11	6	25 max
70 hrs @ 175°C In 7700 Fluid	17	11	10	9	10 max
22 Hrs @ 200°C	17	14	10	9	15 max
168 hrs @ 175°C	26	20	19	15	25 max
336 Hrs @ 200°C	54	57	37	30	
Low Temperature Testing					
TR-10, °C	-31.4	-32.0	-31.8	-31.9	-29°C max
Tg by DSC, °C	-31.2	-32.0	-30.9	-32.5	
Physical Properties @ R.T. - Heat Aged 70 Hrs @ 275°C In Oven					
M-10 , MPa	0.88	0.93	0.8	0.8	
(% Change, M10)	14%	13%	-14%	-4%	
M-100 , MPa	5.0	3.5	6.9	5.2	
(% Change,M100)	-18%	-4%	-34%	6%	
T _B , MPa	11.7	12.1	10.0	10.9	
(% Change,T _B)	-37%	-26%	-35%	-2%	-35% max
E _B , %	180	301	151	252	
(% Change,E _B)	-6%	-3%	-4%	-17%	-10% max
Hardness, A,pts	72	72	73	72	
(Pts Change)	1	1	-2	0	+/- 5
Weight loss, %	7.8	5.2	7.2	6.5	12% max
Physical Properties @ R.T. Aged 70 Hrs @ 23°C In TT-S-735 (III) (Ref. Fuel B)					
M-10 , MPa	0.8	0.78	0.92	0.75	
(% Change, M10)	4%	-5%	-1%	-10%	
M-100 , MPa	6.3	3.5	9.4	4.0	
(% Change,M100)	3%	-6%	-11%	-18%	
T _B , MPa	15.9	12.4	13.3	8.3	
(% Change,T _B)	-14%	-24%	-14%	-26%	-30% max
E _B , %	179	254	156	258	
(% Change,E _B)	-7%	-18%	-1%	-15%	-20% max
Hardness, A,pts	71	70	74	70	
(Pts Change)	0	-1	-1	-2	+/- 5
Vol. Swell, %	1.0	1.6	1.3	1.6	1% – 10%

Appendix II (cont.)

Polymer / filler =	GLT / MT	GLT-S / MT	GLT / Austin	GLT-S / Austin	AMS-R- 83485
Physical Properties @ R.T. - Aged 70 Hrs @ 175°C In 7700 Fluid					
M-10 , MPa	0.5	0.53	0.63	0.52	
(% Change, M10)	-35%	-35%	-32%	-37%	
M-100 , MPa	5.6	3.0	8.3	3.6	
(% Change,M100)	-9%	-19%	-20%	-26%	
T _B , MPa	15.0	14.2	11.2	7.7	
(% Change,T _B)	-19%	-13%	-27%	-31%	-35% max
E _B , %	174	277	152	286	
(% Change,E _B)	-9%	-11%	-4%	-6%	-20% max
Hardness, A,pts	64	65	70	66	
(Pts Change)	-7	-6	-5	-6	+0/-15
Volume Swell, %	16.1	15.9	15.0	14.8	1% to 20%
Physical Properties @ R.T. - Aged 70 Hrs @ 200°C In ARM-200 Fluid					
M-10 , MPa	0.62	0.67	0.69	0.71	
(% Change, M10)	-20%	-18%	-26%	-15%	
M-100 , MPa	5.1	3.0	8.4	3.8	
(% Change,M100)	-18%	-18%	-20%	-24%	
T _B , MPa	15.3	13.5	11.3	8.2	
(% Change,T _B)	-17%	-17%	-27%	-27%	
E _B , %	195	277	146	290	
(% Change,E _B)	1%	-10%	-7%	-5%	
Hardness, A,pts	63	64	69	64	
(Pts Change)	-8	-7	-6	-8	
Volume Swell, %	16.8	15.7	15.3	15.3	

SPECIAL NOTE:

The information set forth herein is furnished free of charge and is based on technical data that DuPont Dow Elastomers believes to be reliable. It is intended for use by persons having technical skill, at their own discretion and risk. Handling precaution information is given with the understanding that those using it will satisfy themselves that their particular conditions of use present no health or safety hazards. Because conditions of product use and disposal are outside our control, we make no warranties, express or implied, and assume no liability in connection with any use of this information. As with any material, evaluation of any compound under end-use conditions prior to specification is essential. Nothing herein is to be taken as a license to operate or a recommendation to infringe on any patents. While the information presented here is accurate at the time of publication, specifications can change. Please check www.dupont-dow.com for the most up-to-date information.

CAUTION: Do not use in medical applications involving permanent implantation in the human body. For other medical applications, discuss with your DuPont Dow Elastomers customer service representative and read Medical Caution Statement H-69237.